VOL. XXVI 1914

TRANSACTIONS

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AMERICAN ELECTROCHEMICAL SOCIETY.

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Twenty-Sixth General Meeting, Niagara Falls,
October 1, 2, 3, 1914.

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TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

157566

VOLUME XXVI

TWENTY-SIXTH GENERAL MEETING

NIAGARA FALLS, OCTOBER 1, 2, 3, 1914

PUBLISHED BY

THE AMERICAN ELECTROCHEMICAL SOCIETY

LEHIGH UNIVERSITY, SOUTH BETHLEHEM, PA.

1915

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VOLUME XXVI. 1914.

TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

PROCEEDINGS

CONDENSED MINUTES OF THE TWENTY-SIXTH GENERAL MEETING OF THE SOCIETY, HELD AT NIAGARA FALLS, OCTOBER 1, 2, AND 3, 1914.

Number of members registered, 119; number of guests, 124; total, 243.

PROCEEDINGS OF WEDNESDAY, SEPTEMBER 30.

Registration opened at 6.00 P. M. at the Clifton Hotel. A meeting of the Publication Committee was held at 7.30, and of the Board of Directors at 8.30 at the Niagara Club.

PROCEEDINGS OF THURSDAY, OCTOBER 1.

The meeting for the reading and discussion of papers was called to order at 9.30 A. M. in the Convention Room of the Clifton Hotel, President F. A. Lidbury in the Chair.

Papers by O. L. Kowalke (abstracted by G. A. Roush), H. Ziegel (abstracted by W. L. Miller), F. H. Getman (abstracted by W. R. Whitney), L. Addicks (abstracted by L. D. Vorce), N. J. Marsh, and H. Schlundt (abstracted by W. F. Bleecker), were presented and discussed as printed in full in these Transactions.

In the discussion of Schlundt's paper the question of the poisonous effects of uranium in the handling of uranium ores was mentioned, and after considerable debate as to whether uranium really had any poisonous effects, the following commit-

tee was appointed to investigate this matter and report to the Society on their findings: W. F. Bleecker, W. R. Mott, A. R. Ledoux (Dr. Ledoux having requested to be relieved from service on this committee, Charles Baskerville was appointed on the committee in his place at a subsequent meeting of the Board of Directors).

In the afternoon a visit was made to Foster's Flats and to the Niagara Gorge.

On the return from Foster's Flats the power plant of the Ontario Power Company was visited.

In the evening at 8.30 a smoker was held in the Convention Room of the Clifton Hotel, while the ladies were entertained in the Palm Room.

PROCEEDINGS OF FRIDAY, OCTOBER 2.

At 9.00 A. M. special cars left the Clifton Hotel for visits to the plants of the International Acheson Graphite Company and the Niagara Alkali Company. The party then divided, some going to the plant of the Spirella Corset Company, and others to the power house of the Hydraulic Power Company.

A session for the reading and discussion of papers was called to order at 2.00 P. M. in the Convention Room of the Clifton Hotel, at which papers by F. J. Tone, C. E. Skinner and L. W. Chubb (presented by Mr. Skinner), C. E. Skinner and L. W. Chubb (presented by Mr. Chubb), W. Lash Miller, F. C. Mathers and B. W. Cockrum (two papers) (presented by Dr. Mathers), and F. C. Mathers, were presented and discussed as printed in full in these Transactions.

At the close of the professional session, the American Cyanamid Company showed moving pictures of the operation of their process at the Cataract Theater.

At 7.00 P. M. dinner was served in the dining room of the Clifton Hotel, followed by an illustrated lecture on the Physical Nature of Color, by C. E. Kenneth Mees, Director of the Research Laboratory of the Eastman Kodak Company.

PROCEEDINGS OF SATURDAY, OCTOBER 3.

The morning session was called to order at 9.30 A. M. at the FitzGerald Laboratories and the paper of Otto Mantius was

presented and discussed as printed in these Transactions. Following this paper the President opened the Symposium on the Practical Side of Electrochemical Investigation, which lasted throughout the day, lunch being served at the Cliff Distributing Company's substation adjoining the laboratory. At the lunch hour, a visit was made to the plant of the Niagara Linen Co. During the course of this Symposium demonstrations were made by F. A. J. FitzGerald (representing FitzGerald Laboratories), L. E. Saunders and J. B. Glaze (representing the Norton Company), A. T. Hinckley (representing National Carbon Company), E. R. Cole (representing International Acheson Graphite Company), C. Dantsizen (representing General Electric Company), Carl Hering (demonstrating the Pinch Effect Furnace), L. W. Chubb (demonstrating the Electropercussive Welding Process), R. S. Wile (demonstrating the Wile Electric Reduction Furnace), P. E. Landolt (demonstrating the Cottrell Process for the Collection of Fumes).

In conclusion it was moved that the sincere and hearty thanks of the Society be extended to the International Acheson Graphite Company, the Niagara Alkali Company, the Spirella Company, the Hydraulic Power Company, the Ontario Power Company, the Cliff Distributing Company, the American Cyanamid Company and the Niagara Falls Linen Company for their courtesies extended in connection with the twenty-sixth meeting of the Society; also to the FitzGerald Laboratories and their staff of assistants who have added a unique and highly instructive day to the program of the meeting; to Mr. C. E. Kenneth Mees for his most interesting lecture; to the naturalist, Mr. H. C. Morphy, for accompanying the excursion to Foster's Flats; to the Niagara Club and the Niagara Falls Country Club for opening their houses and grounds to the visiting members; to the Niagara Falls members, and especially the Chairmen of the Local Committees, and to the Ladies' Committee, for their time and attention so unsparingly given, which have rendered this Twenty-sixth Meeting memorable in the annals of the Society.

The Twenty-sixth Meeting of the American Electrochemical Society was then declared adjourned.

In the evening, an informal rendezvous was held at the Niagara Club, as a farewell to the visiting members.

MEMBERS AND GUESTS REGISTERED AT THE TWENTY-SIXTH GENERAL MEETING

Members.

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Geo. P. Adamson
T. B. Allen
W. H. Arison
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Carl Hering
Alfred Herz
G. K. Herzog
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F. L. Koethen
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J. G. Marshall
E. J. K. Mason
F. C. Mathers
Max Mauran
Ralph McNeill
W. L. Miller
Wm. C. Moore
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J. M. Muir
C. L. Parsons
N. Petinot
J. W. H. Randall
Jos. W. Richards
E. F. Roeber
W. Ross.
A. J. Rossi
G. A. Roush
S. S. Sadtler
E. M. Sargeant
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A. F. Shattuck
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R. Turnbull
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Wm. H. Walker
Walter Wallace
Fred. B. Wheeler
R. H. White
E. H. Whitlock
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A. M. Williamson
J. R. Wilson
C. A. Winder
Charles Wirt
J. A. Yunck

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W. L. Adams, Niagara Falls, N. Y.
Mrs. W. H. Arison, Niagara Falls, Ont.
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L. E. Barton, Niagara Falls, N. Y.
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H. L. Doyle, Niagara Falls, Ont.
C. L. Dunham, Niagara Falls, N. Y.
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John C. Egnia, Niagara Falls, N. Y.
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J. R. MacMillan, Niagara Falls, N. Y.
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W. Judson Marsh, Niagara Falls, N. Y.
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M. E. McDonnell, Altoona, Pa.
W. A. McKnight, Niagara Falls, N. Y.

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Mrs. Chas. Wirt, Germantown, Pa.

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December 31, 1914.

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ASKENASY, Paul, Ph. D. (Feb. 25. '11) Kaiser Allee 20, Karlsruhe (Baden), Germany.
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Walker, W. H.
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Whiting, J.
Winship, Walter

Brookline—
Gravely, J. S.
Howard, H.
Lincoln, E. S.
Ziegel, Henry

South Boston—
Ruppel, H. E. K.

Cambridge—
Jones, Grinell
Lamb, A. B.
Nickerson, W. E.
Richards, T. W.
Yatsevitch, Michael

Dalton—
Newman, M. G.

Great Barrington—
Stanley, Wm.

Lawrence—
Alden, J.

Lynn—
Coates, Jesse
Devers, P. K., Jr.
Kraus, Ernest
Miller, L. B.

Malden—
Berry, E. R.

Newburyport—
Horsch, W. G.

Newton Centre—
Melcher, A. C.

Pittsfield—
Gifford, A. McK.
Kelly, J. F.
Lamker, H. G.
Randolph, C. P.
Slade, W. C.
Tressler, M. E.
Unger, M.
Woodside, F. C.
Wooldridge, W. J.

Roxbury—
Werby, A. B.

Salem—
Hamblet, A. M.

Southbridge—
Oakden, Wm. E.
Ohlwiler, C. H.

Swampscott—
Thomson, E.

Thorold—
Brockbank, C. J.

Walpole—
Manahan, Paul R.

Waltham—
Gegenheimer, R. E.

West Lynn—
Weintraub, E.

Worcester—
Higgins, A. C.
Jeppson, G.N.
Smith, H. B.
Williams, H. J.

MICHIGAN

Ann Arbor—
Bigelow, S. L.
Cole, Harry N.
Patterson, G. W.
Willard, H. H.

Bay City—
Hutchings, Chas. F.

Detroit—
Crosby, E. L.
Diack, A. W.
Jenkins, D. J.
Lane, H. M.
MacGregor, Walter
Marsh, A, L.
Shattuck, A. F.
Stephenson, F. T. F.
Stewart, R. S.

Menominee—
White, A. R.

Midland—
Dow, H. H.

Wyandotte—
Vorce, L. D.

MINNESOTA

Minneapolis—
Frankforter, G. B.
Frary, F. C.
Hunter, J. V.
Lunt, Raymond L.

MISSOURI

Columbia—
Brown, W. G.
Gumaer, P. W.
Schlundt, Herman

Kansas City—
Kent, J. M.

Poplar Bluff—
Randall, Merle

Rolla—
Peterson, C. E.

St. Louis—
Ericson, E. J.
Kohler, H. L.
Queeny, J. F.

MONTANA

Anaconda—
Eagle, Henry Y.
Mathewson, E. P.

Butte—
Bowman, C. H.
Lonergan, P. J.
Silverman, R. B.

Great Falls—
Burns, W. T.
Krejci, Milo W.

Helena—
Gerry, M. H., Jr.
Titus, C. C.

Rexford—
Burwell, A. W.

NEBRASKA

Omaha—
Hall, A. E.

NEVADA

Lovelock—
Reid, John T.

Nelson—
Griffen, H. R.

Reno—
Barton, W. H.

Virginia City—
Symmes, W.

NEW HAMPSHIRE

Berlin—
Barton, C. B.

Durham—
Parsons, C. L.
Perley, G. A.

Keene—
Morgans, F. D.

Wilton—
Abbott, Wm. G., Jr.

NEW JERSEY

Aldene—
Hayes, G. W.

Arlington—
Proctor, C. H.

Belleville—
Hungerford, Oliver T.

Bloomfield—
Amer, H. S.
Franklin, M. W.
Myers, Ralph E.
Romanelli, E.
Spice, Robt.

Carteret—
Breckenridge, J. E.

Chrome—
Deacon, R. W.

East Orange—
Aylsworth, J. W.
Cornell, Sidney
Cowles, Harry D.
Crumbie, Wm. D.
Page, Wm. K.

Elizabeth—
Carrier, C. F.
Pyne, F. R.

Essex Falls—
Robb, Chas. D.

Gloucester City—
Miner, H. S.

Harrison—
Fink, Colin G.
Hart, L. O.
Kraus, Ernest

Haskell—
Cummings, Wm. J.

Highbridge—
Le Boutillier, C.

Hoboken—
Ganz, A. F.

Irvington—
Bachofner, D. K.

Lanford—
Wells, J. S. C.

Jersey City—
Halter, G.
Manger, F. W.
Stillman, T. B.

Maurer—
Alexander, H. H.
Mindeleff, Chas.

Merchantville—
Wickes, C. S.

Montclair—
Crane, F. D.
Ellis, Carleton
Wesson, David

Newark—
Colby, E. A.
Dewey, E. S.
Fernberger, H. M.
Gifford, W. E.
Heath, H. E.
Heimrod, A. A.
Hubley, W. F.
Koerner, W. E.
Liebschutz, M.
Metz, G. P.
Robinson, F. W.
Sievering, Philip
Skidgell, F. M.
Tessler, A.
Van Winkle, A.
Walsh, P. C., Jr.
Weston, E.
Winship, W. E.
Zimmermann, F.

New Brunswick—
North, H. B.
Voorhees, L. A.

Orange—
Cunningham, F. W.
Edison, T. A.
McGall, E.
Smith, H. H.

Perth Amboy—
Aldrich, C. H.
Antisell, F. L.
Bryan, John K.
Emanuel, Louis V.
Fisher, H. W.
Foersterling, H.
Keller, E.
Meyers, H. H.
Nolan, J. J.
Philipp, H.
Renfrew, L. J.
Roessler, F.
Rossi, Louis M.
Sargent, R. N.
Skowronski, S.
Walter, C. A.
Waring, T. D.
Weber, M. G.
Zwingenberger, O. K.

Phillipsburg—
Baker, John T.

Plainfield—
Hibbard, H. D.
Worth, B. G., Jr.

Princeton—
Hulett, G. A.
Northrup, E. F.

Rahway—
Murray, B. L.

Rutherford—
Daft, Leo
Schroeder, C. M. E.

Sewaren—
Buttfield, N. J.
Cowles, A, H.
Lemberg, M.

South Orange—
Yunck, J. A.

Trenton—
Stover, Edward C.

Watchung—
Moldenke, Richard

West Collingswood—
White, A. J.

Westfield—
Fleming, R.

NEW MEXICO

Magdalena—
Benjamin, W. W.

Socorro—
Kemmerer, G.

NEW YORK

Albany—
McElroy, J. F.
Rankin, Herbert E.

Bedford Hills—
Howe, H. M.

Brooklyn—
Cowan, Wm. A
Erhart, W. H.
FitzGibbon, R.
Foster, O. R.
Landolt, P. E.
Maywald, F. J.
Potthoff, Louis
Sheldon, S.
Sperry, E. A.

Buffalo—
Albright, L.
Bierbaum, C. H.
Childs, D. H.
Cooper, K. F.
Corse, Wm. M.
Doty, E. L.
Garretson, Eugene
Mantius, Otto
Onions, Geo. I.
Patch, N. K. B.
Ramage, A. S.
Shepherd, F. A.
Skillman, V.
Wilke, Wm.
Zaremba, Edward

Clinton—
Saunders, A. P.

Glen Falls—
Hilliard, John D.

Hastings-on-Hudson—
Kenyon, O. A.

Ithaca—
Bancroft, W. D.
Bennett, C. W.
Gillett, H. W.
Lohr, J. M.

Kenwood—
Reeve, A. G.

LaSalle—
Sneath, Wm. H.

Lockport—
Howard, L. E.
Kenan, W. R., Jr.

Mamaronech—
Snyder, J. L. K.

Massena—
Doerschuk, V. C.
Falter, P. H.
Hall, H. M.
Herzog, G. K.

Mechanicsville—
Buckie, R. H.

Menands—
Shiverick, M. D.

Monticello—
Drobegg, Dr. Gustave
Isakovics, A. von

Mt. Vernon—
Dabolt, N. E.

New Brighton—
Burger, Alfred

New York City—
Abbé, P. O.
Allyn, R. S.
Aldridge, W. H.
App, O. A.
Baker, H. A.
Barstow, W. S.
Baskerville, C.
Beck, E. A.
Beutner, R.
Bijur, J.
Bjorkstedt, Wm.
Bogue, C. J.
Bowman, W.
Bradley, C. S.
Bradley, W. E. F.
Breneman, A. A.
Brown, H. P.
Browne, D. H.
Browne, deCourcy B.
Buck, H. W.
Caldwell, E.
Cameron, W. S.
Canet, B. Chas.
Carse, D. B.
Case, W. E.
Castle, S. N.
Chandler, C. F.
Clark, W. G.
Clark, W. J.
Cleaves, M. A.
Coho, H. B.
Cohoe, W. P.
Colcord, F. F.
Colvocoresses, Geo. M.
Comstock, L. K.
Corning, C. R.
Crocker, F. B.
Crocker, J. R.
Curran, T. F. V.
Dixon, Jos. L.
Doerflinger, W. F.
Doremus, C. A.
Douglas, James
Dreyfus, W.
Duncan, L.
Dwight, Arthur S.
Eimer, A.
Elliott, A. H.
Emerson, H.
Englehard, C.
EnHolm, O. A.
Eurich, E. F.
Fliess, R. A.
Frank, K. G.
Freas, Thos. B.
Frederick, G. E., Jr.
Fries, H. H.
Gaines, R. H.
Goepel, C. P.
Goodrich, R. R.
Govers, F. X.
Gray, J. H.
Grosvenor, W. M.
Guess, H. A.
Guiterman, K. S.
Halcomb, C. H.
Hansell, N. V.
Harris, J. W.
Hasslacher, J.
Hatzel, J. C.
Hemingway, Frank
Hill, N. S., Jr.
Hirsch, Alcan
Hirschland, F. H.
Hoge, J. F. D.
Huntoon, L. D.
Ingalls, W. R.
Joyce, C. M.
Kennedy, J. J.
Kern, E. F.
Kirchoff, C.
Kissock, Alan
Klipstein, E. C.
Knapp, Geo. O.
Kohn, M. M.
Kunz, G. F.
Langdon, P. H.
Langton, J.
Leavitt, Wm. F. B.
Ledoux, A. R.
Liebmann, A. J.
Lienau, J. H.
Love, E. G.
Lovejoy, D. R.
Lucke, H. J.
MacDonald, J. A.

MacGregor, R. A.
Mailloux, C. O.
Marsh, C. W.
Martin, T. C.
Marvin, A. B.
Mason, E. J. K.
Mason, F. S.
Mastick, S. C.
McAllister, A. S.
McNeill, R.
Mershon, R. D.
Merton, R.
Metz, H. A.
Moody, H. R.
Morgan, J. L. R.
Morse, W. S.
Muir, J. M.
Muschenheim, F. A.
Nathan, A. F.
Nichols, W. H.
Nichols, W. S.
Ornstein, G.
Pearson, F. S.
Pennie, J. C.
Pleiss, Paul
Price, E. F.
Prindle, E. J.
Prosser, H. A.
Ricketts, L. D.
Riglander, M. M.
Riker, J. J.
Roeber, E. F.
Rossi, A. J.
Ruhl, L.
Schildhauer, Edw.
Schloss, J. A.
Schuetz, F. F.
Scudder, H.
Seward, G. O.
Sharp, C. H.
Sieger, G. N.
Slocum, H. J.
Smith, Dyer
Smith, E. A. C.
Speiden, C. C.
Spilsbury, E. G.
Statham, Noel
Stone, Geo. C.
Stone, I. F.
Stone, J. P.
Stoughton, Bradley
Swenarton, W. H.
Takamine, J.
Taylor, J. R.
Teeple, J. E.
Thatcher, C. J.
Thompson, J.
Thompson, R. N.
Toch, M.
Tucker, S. A.
Van Arsdale, G. D.
Waddell, M.
Waldo, W. B.
Walker, A. L.
Waterman, F. N.
Weeks, F. D.
Weinstein, L. J.
Whitaker, M. C.
White, J. G.
Wiechmann, F. G.
Wigglesworth, H.
Williams, A.
Williams, J. T.
Witherell, C. S.
Wood, E. F.
Wright, Arthur
Zeller, R.

Niagara Falls—

Arison, W. H.
Barnes, Carl S.
Barton, P. P.
Bayard, R. A.
Becket, F. M.
Beckman, J. W.
Bennie, P. McN.
Bliss, Wm. Lord
Callahan, J. F.
Carveth, H. R.
Cole, E. R.
Converse, V. G.
Cox, G. E.
Dunlap, O. E.
Edmands, I. R.
FitzGerald, F. A. J.
Fowler, R. E.
Giles, I. K.
Glaze, John B.
Griffith, J. R.
Hall, C. M.
Harper, J. L.
Haskell, F. W.
Hendrie, G. A.
Hepburn, D. M.
Hinchley, A. T.
Horry, W. S.
Hooker, A. H.
Humbert, E. P.
Hutchins, Otis
Imlay, L. E.
Johnson, J. A.
Kellogg, H. W.
Kemmer, F. R.
Koethen, F. L.
Leffel, C. E.
Lidbury, F. A.
MacMahon, J.
MacMillan, J. R.
Marshall, J. G.
Mauran, M.
McCullough, H. F.
Meredith, W. F.
Moritz, C. H.
Moyer, G. C.
Petinot, N.
Phillips, R.
Randall, J. W. H.
Ross, Wm.
Russell, C. A.
Saunders, L. E.
Sergeant, E. M.
Simmers, A. L.
Smith, A.
Smith, E. S.
Snowdon, R. C.
Speiden, E. C.
Sprague, E. C.
Stamps, F. A.
Stone, G. W.
Taylor, Chas. E
Tone, F. J.
Vaughn, C. F.
Wallace, W.
White, R. H.
Wilcox, W. G.
Wilson, J. R.
Williamson, A. M.
Winder, C. A.

Ossining—

Acker, Chas. E.

Penn Yan—

Taylor, E. R.

Piermont—
Main, W.

Poughkeepsie—
Moulton, C. W.
Quintard, Edw.

Prince Bay (S. I.)—
Johnston, F. A.
Johnston, W. A.

Richmond Hill (L. I.)—
Haslwanter, C.
Herreshoff, J. B.
Maeulen, F.
Smull, J. G.

Rochester—
Hutchings, J. T.
Schwarz, R. C.

Schenectady—
Andrews, W. S.
Arsem, W. C.
Arthur, Walter
Berg, E. J.
Capp, J. A.
Coffin, F. P.
Coolidge, Wm. D.
Creighton, E. E.
Dushman, Saul
Ferguson, C. V.
Hansen, C. A.
Hawkins, L. A.
Lamar, M. O.
Langmuir, I.
MacKay, G. M. J.
Murphy, E. J.
Reist, H. G.
Rushmore, D. B.
Seede, J. A.
Steinmetz, C. P.
Taylor, J. B.
Valentine, I. R.
Van Brunt, C.
Whitney, W. R.
Willey, L. M.

Sea Gate—
Rowand, L. G.

Syracuse—
Brookfield, W. B.
Matthews, J. A.
Pennock, J. D.

Troy—
Hunter, M. A.
Lincoln, A. T.

Watertown—
Williams, F. M.

Williamsville—
Cushing, H. M.

Yonkers—
Baekeland, L. H.
Harrington, E. I.

NORTH CAROLINA

Charlotte—
Gilchrist, P. S.

Clemmons—
Pickens, Rufus H.

Stackhouse—
Betts, A. G.

West Raleigh—
Browne, W. H.

Whitney—
Marseilles, Wm. P.
Rockwell, Wm. F.

OHIO

Akron—
Shaw, E. C.

Alliance—
Baily, T. F.
Bird, C. T.
Cope, F. T.

Amherst—
Briggs, F. H.

Cincinnati—
Ashton, James

Cleveland—
Brady, W. B.
Brown, J. W.
Chillas, R. B., Jr.
Clymer, W. R.
Cook, Edw. B.
Crider, J. S.
Fleming, S. H.
Graves, W. G.
Holmes, M. E.
Hudson, A. J.
Hyde, E. P.
Koehler, W.
Mainwaring, Wm. D.
Mann, W. W.
Pratt, E. B.
Smith, A. W.
Taylor, F. D.
Wellman, S. T.
Whitlock, E. H.
Woodward, J. M.

Columbus—
Flowers, A. E.
Withrow, J. R.

Dayton—
Clements, F. O.
Deeds, E. A.
Dorsey, H. G.
Foley, Chas. B.
Niswonger, E. E.

Fostoria—
Downes, A. C.

Fremont—
Goodwin, J. H.
Ziegler, W. W.

Lakewood—
Bullock, A. R.
Chaney, N. K.
Moore, Wm. C.
Mott, W. R.
Ordway. D. L.
Pulman, O. S.
White, F. L.

Middletown—
Ahlbrandt, G. F.

Napoleon—
Long, G. E.

Oberlin—
Crafts, W. N.

Rittman—
Laib, Walter

Salem—
Davis, D. L.

Steubenville—
Homan, J. G.

Warren—
Richardson, E. A.

Youngstown—
Brinker, H. L.
Danforth, Chas. W.

OREGON

Cornucopia—
Keeney, R. M.

PENNSYLVANIA

Altoona—
Casselberry, H.
Hanger, S. R.

Ambler—
Coleman, Wm. B.

Ambridge—
Farnham, F. F.

Aspinwall—
Hessom. B. F., Jr.

Bethlehem—
Dyrssen, W.
Fehnel, J. Wm.

Brackenridge—
Connell, H. R.
Ober, J. E.

Braddock—
Friedlaender, E.
Lewis, J. D.

Bridgeville—
Saklatwalla, B. D.

Bryn Mawr—
Ely, T. N.
Getman, F. H.

Canonsburg—
Allen, T. B.
Bleecker, W. F.
Guerber, A. J.
Morrison, W. L.

Chester—
Comey, A. M.

Coraopolis—
Stafford, S. G.
von Kugelgen, F.

Creighton—
Gelstharp, F.
Kerr, Chas. H.
Parkinson, J. C.

Crofton—
Fawcett, L. H.

Duquesne—
Unger, J. S.

Easton—
Adamson, G. P.
Gordon, C. McC.
Hart, E.
Shimer, E. B.

Ellwood City—
Barton, Chas. R.
Dunn, J. J.
Offutt, J. W.

Erie—
Diller, H. E.

Ford City—
Gildard, W. R.

Gettysburg—
Parsons, L. A.

Harrisburg—
Baltzell, W. H.
Calder, A. R.

Ingram—
Armor, J. C.

Johnstown—
Appel, M.
Kenney, E. F.
Parkhurst, C. W.

Latrobe—
Sessions, R. L.

Lebanon—
Crowell, W. J., Jr.
Weimer, E. A.

McKeesport—
Clarke, E. B.
Garratt, Frank
Goodspeed, G. M.

Munhall—
Hopkins, G. A.
James, E. T.
McNiff, G. P.
Reese, P. P.

New Castle—
Samuels, Wm. P.

New Kensington—
Wilson, I. W.

Palmerton—
Holdstein, L. S.

Parnassus—
Blough, Earl

Philadelphia—
Benoliel, S. D.
Bonine, Chas. E.
Bower, F. B.
Breed, G.
Brophy, O.
Cary, C. R.
Chance, H. M.
Clamer, G. H.
Corbin, J. R.
Davis, F. W.
Davis, R. W., Jr.
Devereux, W.
Eglin, W. C. L.
Fraley, J. C.
Furness, R.
Gerety, J. J.
Gibbs, A. E.
Goldbaum, J. S.
Hering, C.
Hess, H.
Hicks, Edwin F.
Hitchcock, F. R. M.
Hornor, H. A.
Howard, G. M.
Keith, N. S.
Kitsee, I.
Lafore, J. A.
Lavino, E. J.
Lay, J. Tracy
Luckens, H. S.
McConnell, J. Y.
McFarlin, J. R.
Merzbacher, Aaron
Meyer, J.
Morgan, Leonard P.
Nodell, Wm. L.
Paul, H. N.
Rosengarten, G. D.
Russell, C. J.
Sadtler, S. P.
Sadtler, S. S.
Salom, P. G.
Schamberg, M.
Smith, E. F.
Smith, E. W.
Snook, H. C.
Stevens, J. F.
Taggart, W. T.
Thwing, C. B.
Weeks, C. A.
Weisenburg, Andrew
Weitlaner, R. J.
Wirt, Chas.

Pittsburgh—
Bacon, R. F.
Bennett, B. F.
Brown, John T., Jr.
Connell, W. H.
Coyle, J. A.
Crabtree, F.
Davis, S. A.
Dewey, B.
Donkin, Wm. A.
Dougherty, J. W.
Flannery, Jas. J.
Gibson, C. B.
Goodale, S. L.
Hartley, R. H.
Hitchcock, H. K.
James, J. H.
Kemery, P.
Kier, S. M.
Laughlin, H. H.
Lawrence, J. N.
Lincoln, P. M.
McDonald, R. A.
McKinley, Jos.
Murphy, D. H.
Orr, R. S.
Osborne, L. A.
Page, G. S.
Pinkerton, A.
Pope, Chas. E.
Ramsey, F. H.
Ray, H. C.
Rodman, H.
Sargent, G. W.
Schluederberg, C. G.
Slocum, F. L.
Snelling, W. O.
Speller, F. N.
Stewart, R. T.
Storey, O. W.
Stupakoff, S. H.
Tiemann, H. P.
Uhlenhaut, F., Jr.
Vogt, C. C.
Webb, Z.
Wiley, B.

Primos—
Boericke, G.
Stein, W. M.

Ralston—
Dalburg, F. A.

Reading—
Parker, Jas. H.

Roaring Springs—
McDonald, F.

Scranton—
Bryden, C. L.

Sewickley—
Wadsworth, F. L.

Sharon—
Kranz, W. G.

South Bethlehem—
Buck, C. A.
Callen, A. S.
Fischer, Siegfried
Franklin, W. S.
Lomax, C. S.
MacNutt, B.
Richards, J. W.
Roush, G. A.
Seyfert, S. S.

State College—
Dudley, Boyd, Jr.
Pond, G. G.
Turnock, L. C.

Steelton—
Carney, F. D.
Koch, S. B.
Reed, J. C.

Swarthmore—
Alleman, G.

Tyrone—
Fernau, H. C.

West Pittsburgh—
Buch, N. W.

Wilkinsburg—
Skinner, Chas. E.

York—
Schleeder, L. B.

RHODE ISLAND

Greene—
Perry, C. M.

Newport—
Richards, E.

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Hagge—
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A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 1, 1914, President F. A. Lidbury in the Chair.

THE SOURCES OF IMPURITIES IN CATHODE COPPER.

By Lawrence Addicks.

The grossly disproportionate effect of mere traces of certain impurities on the physical properties of pure metals is most striking. The older methods of refining in metallurgy were generally based upon some chemical reaction carried on while the impure metal was in the fluid state, with the result that as the amount of impurities became small, the speed of reaction diminished to such an extent that the operation could commercially yield but a moderately pure metal. For this reason the products of certain mineral districts where the raw material happened to be unusually pure, obtained a special reputation. This was notably so in the case of copper where the relatively pure native copper ores of Michigan yielded a superior product for many years, so that Lake copper commanded a premium in the market long after the reason for it had disappeared.

Electrolysis has practically displaced other methods for refining copper, silver and gold, and has found special fields in the metallurgy of nickel and lead, with iron yet to follow. The reason is found in the great purity of the product. The older methods endeavored to take the impurity out of the metal; electrolysis takes the metal out of the impurity and secures mass action as an ally instead of as an enemy. In addition, elements which before were but troublesome impurities, have been changed to valuable by-products.

The electrolytic refining of metals in general and of copper in particular, consists of taking as an anode, material which has been already subjected to a brief fire refining and leaving the remaining impurities partly in the electrolyte and partly as insoluble slimes, giving a triple separation of the elements contained in the anode. As electrolysis is justified by the purity of the cathode, a careful study of the sources of contamination of this product during electrolysis, slight as this is, is of great importance to the refiner.

Several years ago, I collected a large quantity of data in the endeavor to ascertain what rôle electrolyte included within the pores of the cathode body played in contamination, and through the courtesy of those in charge, many figures from the Raritan, A. S. & R., Balbach, Great Falls and Buffalo refineries were added to those available at the Chrome plant with which I was especially concerned. The study proved to be too complex to yield conclusive results, but a number of relations were established, which may be of interest to others.

Impurities in refined copper may (*a*) cause a low electrical conductivity; (*b*) result in brittleness or weakness; or (*c*) be a source of loss of by-products of value. The first class is represented by phosphorus, aluminium, silicon, arsenic and antimony; the second by bismuth, lead, tellurium and selenium; and the last by silver, gold, platinum and palladium. It is further interesting to note, that class (*a*) forms with copper alloys of excellent mechanical properties, while class (*b*) is practically insoluble in copper, forming a brittle matrix around the copper grains in the cast bar.

From the refining point of view impurities may be due to (*d*) electrolytic deposition; (*e*) inclusion of electrolyte in the crystalline structure of the cathode; (*f*) mechanical contamination from anode slimes suspended in the electrolyte and attaching themselves to the cathode; and (*g*) substances entering during melting from furnace, fuel and tools, and from other outside sources. It must be remembered that the finished copper is guaranteed to run at least 99.88 percent Cu, that it commonly does run 99.93 percent in the melted bar and that it is very unusual to find 0.01 percent of any one impurity except oxygen, probably as cuprous oxide in solid solution, or possibly some other dissolved gas, such as argon or carbon monoxide. The copper content specification is intended to control this gas content, which is innocuous within these limits, while a conductivity guarantee imposes a very strict limit upon the impurities of class (*a*). Class (*b*) is ignored in specifications because these elements are but very rarely present in undesirable quantities and those who have studied the question longest do not feel confident to prescribe any definite limits, on account of the compensating action of different impurities upon one another. Class (*c*) does no harm in quantities a refiner can

possibly afford to let go out, as but very small amounts of these elements need be present to pay for a re-refining.

We may prepare a table of the possible sources for each impurity commonly found in refined copper about as follows:

TABLE I.

Impurity	Slimes	Included	Deposited	Outside Sources
Arsenic	Yes	Yes	Possibly	No.
Antimony	Yes	Slight	Possibly	Yes
Iron	Slight	Yes	No.	Yes
Zinc	No	Yes	No.	No.
Nickel	Yes	Yes	No.	No.
Lead	Yes	No	No.	Yes
Selenium	Yes	No	No.	No.
Sulphur	Yes	Yes	No.	Yes
Tellurium	Yes	No	No.	No.
Silver	Yes	No	No.	No.
Gold	Yes	No	No.	No.

In examining this table wherever we have three "noes" and one "yes" we have a direct measure of the contamination from the source marked "yes." Silver, gold, selenium and tellurium give direct results for slimes contamination in this way. Applying the factor thus found for slimes, we should be able to eliminate that column and examine the other three separately. Zinc should give a measure of inclusion in the same way, narrowing the columns to two. By exclusion we should be able to make a complete separation. Actually we find much conflicting evidence. This is doubtless due largely to the fact that the data on the copper are obtained after melting and the slagging and volatilization in the refining furnace is sufficient to upset the ratios with which we are dealing.

The classification shown in the table is based first on the known analysis of anode slimes. It is evident that any element present in the slimes must be entered as "yes" under that column. In the same way any element dissolved in the electrolyte must be entered as "yes" under "included." "Deposited" is open to some question. Obviously an element not dissolved in the electrolyte can not be deposited. Of course, if conditions become so abnor-

mal that silver sulphate or selenious acid is present in the electrolyte the corresponding entries will have to be changed. In the same way if the circulation of the electrolyte is defective, we may have voltages acting which will undoubtedly deposit arsenic and other impurities. The only danger is that, when considering average figures from large-scale operation, we may include sufficient abnormal conditions to vitiate the result.

In addition to the "outside" influence of ratios disturbed by melting, we have several definite interferences. The tanks are lined with antimonial lead and as this sulphates and to a certain extent flakes off, we have artificial slimes introduced. Then the iron of the rabbles and blow pipes used in the refining furnace enters the bath to a considerable extent. The products of combustion and ash from the coke and charcoal with which the bath is covered introduce sulphur. The fouling with lead and antimony is very small, but iron and sulphur are large items.

TABLE II.

Plant	Current Density Amperes Per Sq. Ft.	Current Density Amperes Per Sq. M	Percent Ag in Anodes Lost in Cathodes	Percent Au in Anodes Lost in Cathodes
A	12	129	(1.00)	.... Very low Ag in anodes
B	16	172	0.52	0.56
C	16	172	0.71	0.68
D	20	215	0.40	1.60 Rough figures
E	20	215	0.77	(0.26) Low Au method
F	35	377	2.25	(1.60) Probably low Au method

Examining the case as it stands, however, we may start with silver and gold. Here abundant information is available, as the metal losses are always very closely watched. A very puzzling case developed, in that at two refineries the proportionate silver losses ran about twice the gold. A careful investigation brought out the fact that at one of them and probably at the other, the assay method used gave low results for gold on account of the very small quantity present and correcting this would doubtless bring the gold up to the silver figures. As the silver and gold in the anodes all go into the slimes, the cathode losses should be directly proportional to the anode contents, assuming an equal

amount of float slimes in the different refineries. With high current densities there is more disturbance in the tank, due partly to increased circulation, than when low density is used. We should therefore expect increased percentage losses at plants where high current densities are obtained.

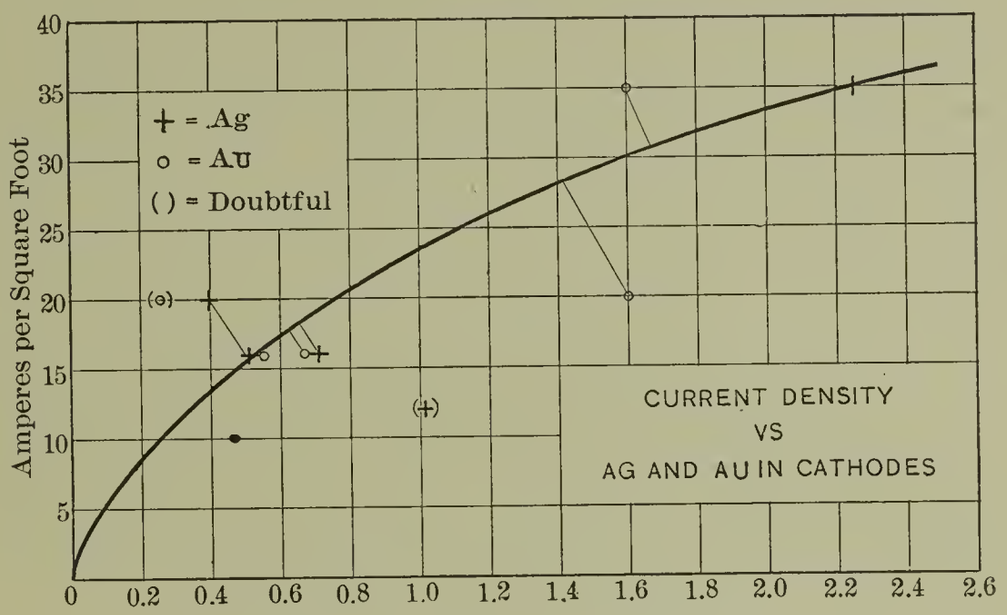

FIG. 1.

An inspection of Fig. 1 shows that current density has directly or indirectly a marked effect on the amount of slimes reaching the cathodes. Also that for densities between 15 and 20 amperes per sq. ft. (172-215 amp, per sq. m.), comprising most of the plants (the data in this paper are all from multiple-system plants; series plants are complicated by fouling of cathodes from adhering anode scrap) some 0.7 percent of the silver and gold in the anodes adheres as slime to the cathodes, or in other words, the efficiency of refining is about 99.3 percent. As the silver and

TABLE III.

Element	Percent in Slimes	Percent in Cathodes	Percent Slimes X 0.000154	Remainder	Percent Fouling by Slimes
Ag	33.82	0.00520	0.00520	0	100
Te	2.74	0.00023	0.00042	Negative	100
As	2.77	0.00105	0.00043	0.00062	41
Sb	5.40	0.00255	0.00083	0.00172	32
Fe	0.19	0.00330	0.00003	0.00327	1
Ni	4.98	0.00520	0.00077	0.00443	15
Pb	1.86	0.00070	0.00029	0.00041	41
S	2.01	0.00076	0.00031	0.00045	41

gold in the anodes all goes into the slimes, this is the same thing as saying an equivalent amount of the anode slimes is carried over to the cathodes and knowing the analysis of the slimes, we should be able to figure the degree of fouling from this source. It will be of interest to test an actual case as in Table III.

The case taken is one where the fouling as shown by the silver is twice normal, which, however, is of no harm as an example. Figures happen to be available for this period (which covers many million pounds of cathodes) for raw slimes, that is slimes that have simply been boiled free of soluble sulphates before sampling, whereas slimes are nearly always roasted and boiled in dilute sulphuric acid until practically copper-free before samples are taken and this operation somewhat disturbs the balance of impurities. The figures show clearly that except in the case of the insoluble group of elements before mentioned the slimes are not the only source of fouling. Except for iron and sulphur, the melting operation may slightly reduce impurities, but cannot increase them, so our argument is on the safe side.

The position can be attacked, however, on the ground that silver and gold are heavy and sink readily to the bottom of the tank, while the lighter oxidized impurities chiefly make up the float slimes. Emrich, in his paper on the Refining of Blister Copper, presented before the American Institute of Mining Engineers, in 1904, argues backwards from the cathode analysis to prove this point and concludes that practically all the fouling comes from float slimes. I think that some of the data submitted later shows that his position is not sound. It is true that material collected at some distance away in launders, etc., runs disproportionately low in gold and silver, but this is truly floating material, whereas the cathode fouling takes place but an inch from the anode and the bottom of a cathode is known to be more foul than the top.

It may also be well to point out here that satisfactory samples cannot be obtained by drilling cathodes. In the first place individual cathodes are irregular in purity, but more important is the fact that such samples always show too low results for impurity. This is due to the fact that adhering slimes are usually the nuclei of nodules forming rough deposits and it is necessary to take a relatively smooth part of the cathode in order to drill a hole. Nodules broken off and melted will often show ten times the

impurities contained by a drilled sample of the body of the cathode. For these reasons it is necessary to fall back on melted samples or fire-refined cathodes in spite of the accompanying disadvantages, when undertaking work of this character.

The next element on which abundant data are available is arsenic, the ancient, but long since subdued, enemy of copper

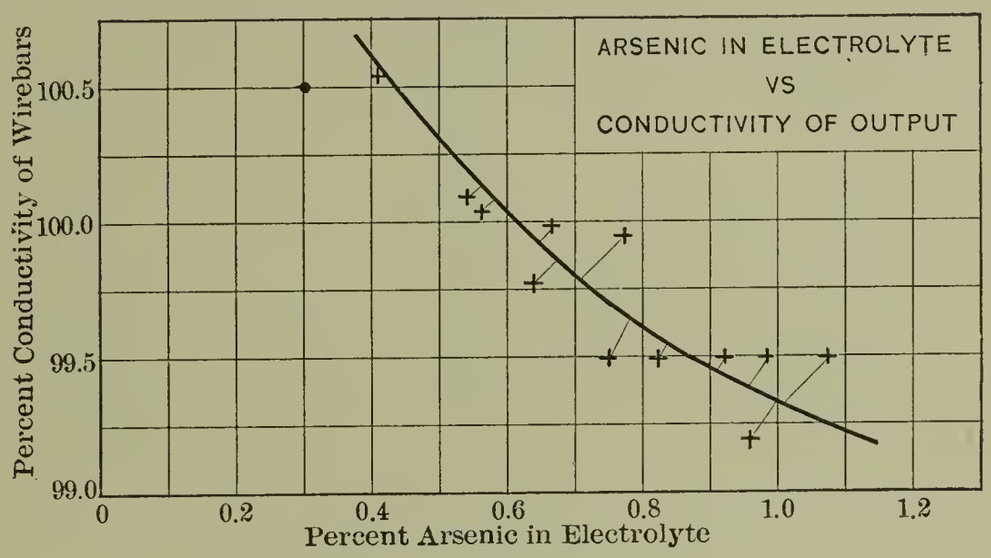

FIG. 2.

refining. It was early found that the presence of arsenic in an electrolyte was detrimental to the conductivity of the cathodes. Table IV gives the average monthly value of arsenic in the electrolyte and the conductivity of the wire bars of one of the refineries for a year.

TABLE IV.

Month	As in Electrolyte	Conductivity of Wirebars
January	0.83	99.47
February	0.95	99.34
March	0.99	99.54
April	1.06	99.50
May	0.91	99.59
June	0.76	99.48
July	0.67	99.99
August	0.56	100.05
September	0.64	99.84
October	0.78	99.90
November	0.55	100.12
December	0.42	100.60

These figures are plotted in Fig. 2, which clearly shows their correspondence.

The same relation can be established by direct comparison of arsenic in electrolyte and in wire bars. In Table V, figures over a long range of time and for several plants have been grouped, in some cases by periods and in some by classes.

TABLE V.

Plant	Representing	Percent as in Electrolyte	Percent as in Wirebars
A	1909	0.728	0.0020
A	1910	0.789	0.0016
A	1911	0.506	0.0018
A	1912	0.219	0.0019
A	1913	0.194	0.0015
B	21 Months	1.04	0.0016
C	1 Lot	0.25	0.0016
C	5 Lots	1.64	0.0025
C	4 Lots	2.37	0.0033
C	3 Lots	3.55	0.0041
D	25 Months	1.34	0.0018

These figures plotted in Fig. 3 indicate an arsenic content in the wire bars of 0.0013 percent when there is no arsenic in the electrolyte, which should be a measure of the fouling due to slimes. Anything above 0.0013 percent would be due to either

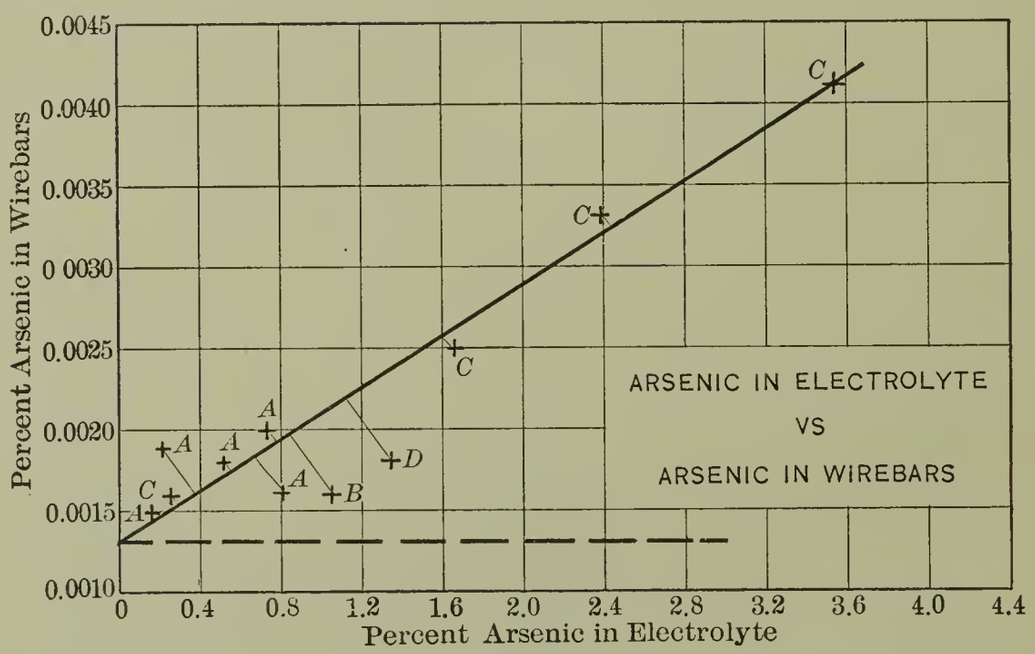

FIG. 3.

inclusion of electrolyte or electrolytic deposition of arsenic. Included electrolyte should vary directly with the composition of the solution, other things being equal. On the other hand electro-

deposited arsenic should increase with the voltage. Now plant C runs at very high current density and therefore at correspondingly high voltage, but no corresponding increase in arsenic in the wire bars can be noticed.

The figures, therefore, might seem to indicate inclusion as the chief source of fouling above the slimes minimum. These Great Falls figures (plant C), by the way, have since been summarized in a paper by Mr. W. T. Burns, Superintendent of that refinery, presented before the American Institute of Mining Engineers last year.

We may now turn to nickel for further light on this subject. Here we have an element whose sulphate has a very high decomposition voltage and it would seem unlikely that any nickel at all, regardless of the voltage, could be electro-deposited from a strongly acid solution. Table VI gives results from nickel from various sources.

TABLE VI.

Plant	Representing	Percent Ni in Electrolyte	Percent Ni in Wirebars
A	1909	1.95	0.0041
A	1910	1.60	0.0055
A	1911	1.73	0.0066
A	1912	1.32	0.0038
A	1913	1.78	0.0037
B	10 Months	0.973	0.0045
C	Rough figures	1.00	0.0016
D	1 Lot	0.594	0.0014

These figures when plotted in Fig. 4 indicate a straight line relation, as in the case of arsenic, but with no nickel in the cathodes when nickel is absent from the electrolyte. In other words there is no fouling from the slimes. This may easily be so, as the slimes usually carry much less nickel than shown in Table III and even there the possible fouling is but 15 percent. Further, this nickel is probably present as nickel oxide which is slowly soluble in the electrolyte and float slimes may be largely cleansed of their nickel by the electrolyte. The surprising thing, however, is the large amount of nickel found in the cathodes. While a rise of 1 percent in the content of the electrolyte shows in Fig. 3 a corresponding rise in the wire bars of but 0.0009

percent, in the case of nickel it shows 0.0028 percent, or three times as much. Emrich found the same thing. He says: "The large difference shown between the calculated and the observed values of nickel in the cathodes, is a mystery which I have been prevented from investigating."

Either a large quantity of arsenic is removed in the refining furnace, or we must accept electrodeposition of nickel, as the included electrolyte must be rigidly equivalent in the two cases. Those who have tried eliminating traces of arsenic in a refining furnace, except by the use of lime and soda ash or similar fluxes, which is done only in special cases where undesirable quantities

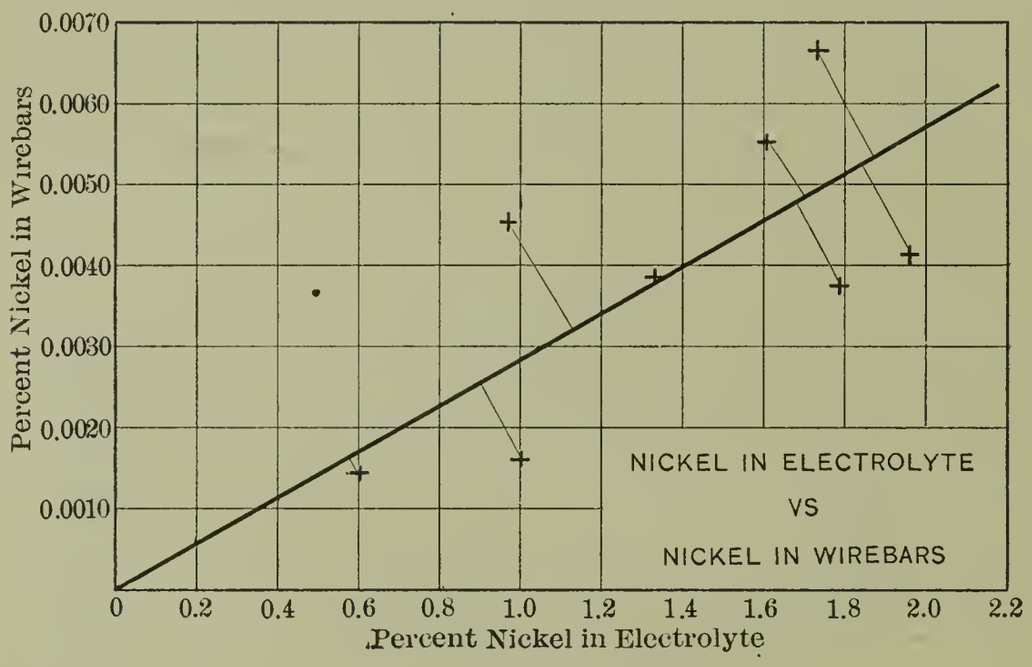

FIG. 4.

of arsenic are present, will lean toward the nickel deposition explanation.

Some laboratory experiments indicated that while no nickel can be deposited from a pure acidulated solution of nickel sulphate, traces can be found in the cathodes when arsenic is added to the electrolyte, which suggests some ion based on the known affinity of nickel for arsenic. Also in an electrolytic separation of copper and nickel by the Hybinette process where the electrolyte was very high in nickel sulphate, copper containing as much as 0.1 percent nickel has been produced. While these conditions are far from those obtaining in a copper refinery, the results greatly strengthen the deposition argument.

Finally we can examine the inclusion argument on the basis of the specific gravity of cathode copper. If from Fig. 3 a cathode runs 0.0022 percent, minus 0.0013 percent (due to slime) equals 0.0009 percent arsenic due to inclusion of electrolyte running 1.0 percent arsenic, the cathode would contain 0.0009 times 100 or 0.09 percent of electrolyte of a specific gravity of let us say 1.15. As copper has a specific gravity in the neighborhood of 8.9, the amount present by volume would be 0.09 times 8.9 divided by 1.15, or 0.7 percent, which is a measure of the porosity of the cathode. The specific gravity of the copper would be correspondingly diminished, but the amount would be so slight that the known facts do not contradict it. Cathode copper actually shows a specific gravity of about 8.9 similar to worked cast copper, but on the other hand we know that cathode copper is very hard—experimental work on direct production of electrolytic wire has given considerable trouble from wear on the dies—and we should therefore expect to find it denser than any other copper if it were absolutely non-porous.

We therefore arrive at about where we started, believing that slimes, included electrolyte and direct deposition all have a hand in the fouling of cathodes, but without being able to assign definite values to each. The relation between arsenic and nickel in electrolyte and in wire bars does seem to be reasonably established and this is of some practical value.

I wish to acknowledge my indebtedness to Messrs. H. H. Alexander, F. L. Antisell, W. T. Burns, R. W. Deacon, Geo. L. Heath, W. L. Spalding and C. S. Witherell for furnishing a large share of the mass of data on which the original study was based.

DISCUSSION.

W. D. Bancroft (*Communicated*): The fact that nickel is deposited with the copper to an extent increasing with the concentration is probably due to the fact that copper and nickel form a continuous series of solid solutions. Ashbrook[1] found that nickel was precipitated with copper in rapid electrolytic analysis.

[1] Jour. Am. Chem. Soc., 26, 1287 (1904).

W. R. WHITNEY: Papers such as Mr. Addicks' are exceedingly important to members of the Society. They point to the practicability of further improvements in the quality of copper. It is worth while using the best obtainable. In most electrical apparatus copper of 98 percent conductivity, Mathiessen standard, is being accepted. The metal has been refined to a value of 101.8 conductivity. As the dimensions and cost of electrical apparatus depend on the conductivity of the copper there ought to be a demand for the highest grade.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 2, 1914, President F. A. Lidbury in the Chair.

ON THE FORMATION OF A BADLY CONDUCTING FILM ON COPPER ANODES IN COPPER CYANIDE SOLUTIONS.

By W. Lash Miller.

While preparing some experiments for the laboratory of the Toronto Branch of the American Electroplaters' Society, a current of about one ampere was sent through a copper cyanide bath (No. 3, below) using strips of copper 9 cm. x 3 cm. as electrodes, the 110 volt D. C. lighting circuit as source of current, and a 32 c. p. carbon filament lamp as resistance in the circuit. The lamp, bright at first, within a minute's time burned dim, the evolution of hydrogen at the cathode fell off, and a voltmeter set over the cell showed a potential difference of 45 volts. On examination, the anode was found to be coated with a smooth yellow-green adherent deposit; and a few simple trials soon showed that the resistance of this film was the cause of the drop in current.

The formation of such a deposit evidently sets an upper limit to the current density at the anode that can be used efficiently with any given bath; a number of solutions recommended in the literature, which had been made up for another purpose, were tested from this point of view.

It was soon found, as expected, that the current density at which a noticeable film is formed depends on the shape and position of the anode and on the amount of circulation in the electrolyte; the simple apparatus shown in Fig. 1 was then constructed, and the various electrolytes were compared under the conditions obtaining in it.

In the figure, *A* is the anode, a cylinder of copper rod 2 cm. in diameter, *C* is the cathode, a hollow cylinder of sheet copper 2 mm. thick, 4 cm. internal diameter, slit down one side so that the height of the liquid could be read on a paper scale *S* pasted on the outside of the glass container *G*. Anode and cathode were

held in position by means of two corks, of which the lower, *L*, was provided with a socket to catch the end of the anode and with notches around its edge to let the solution flow out evenly when the tap was opened. The zero mark of the scale was level with the upper side of the lower cork, its graduations ran from 1 to 10, and gave the area of submerged anode in tenths of a square decimeter; division 10 was 2 or 3 centimeters below the upper cork, *U*.

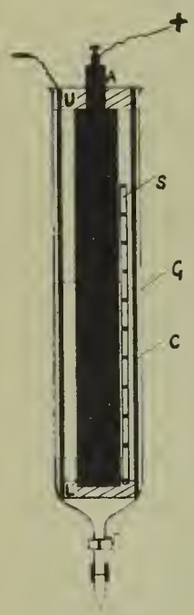

FIG. 1.

To ensure that the liquid would run out of the cell at the same rate in different experiments, a glass nozzle was attached by rubber below the tap, and the latter was always either closed or wide open; with the nozzle used it took the solutions 60 seconds to fall from division 10 to division 1 on the scale, the volume comprised between these two marks being 197 cc.

Before each measurement, anode and cathode were cleaned by nitric acid, washed under the tap, and placed in the container; 250 cc. of an electrolyte was poured in through the funnel, the tap was opened, and as the surface passed division 10, the current (about one ampere) was switched on and its amount recorded. As the level fell, a voltmeter[1] wired across the cell was read; at first the voltage was low, then suddenly began to rise, often with a well marked "jump," whereupon the circuit was broken, the tap closed, and the level recorded; hence the current density when the sudden rise in voltage began was calculated. If the tap were left open, and the current on, the voltage rose rapidly until the needle reached the end of the scale.

Each measurement was made in duplicate, and when it appeared that the initial current was too high, the work was repeated using half an ampere; with baths 5, 11 and 13 a still smaller current would have been better.

The amount of "jump" and the rapidity of the rise in voltage varied from case to case; in every case examination of the anode showed that a precipitate had been formed there, but the color of the deposit varied from yellow to green and brown, and its appearance from smooth and coherent to streaky, depending on the composition of the electrolyte.

[1] Reading from 0 to 3 volts, graduated to one fiftieth volt.

The following table gives the compositions of the solutions employed; the last three columns give the number of grams of "free cyanide" per litre determined as recommended by Langbein, with volumetric silver nitrate and sodium chloride as indicated—the current density in amperes per square decimeter at the anode when the voltage jumps occurred, and the result of the "copper titration" described below.

Bath No.	Composition of Bath						Free Cyanide	Current Density	Copper Titration
	Copper salt used	Grams per litre Cu.	Grams per litre KCN	Grams per litre $NaHSO_3$	Grams per litre K_2CO_3	cc. of NH_4HO 0.88 s.g. per litre			
1	Sulphate	12.5	12.5	0	0	55	80.0	3.0	33.0
9	Sulphate	4.6	53.5	0	S* 72	0	36.0	1.75	22.6
8	Carbonate	27	100	25	30	0	43.5	1.33	18.0
15	.....	23	....	.....	.....	....	8.0	1.3	16.2
3	Acetate	14	53.3	44	S* 53	0	10.5	1.25	13.3
7	Carbonate	20	75	15	15	0	28.0	1.25	13.0
4	Carbonate	14	53.5	0	0	0	15.5	1.1	7.7
14	.....	9	....	.....	.....	....	13.8	1.0	6.8
6	Sulphate	9.5	38	0	0	26	16.0	1.0	4.3
2	Sulphate	6.5	21.4	18	S* 48	0	6.3	1.0	4.0
13	.....	45	....	.....	.....	....	6.0	0.5	2.2
11	Acetate	5.1	20	0	0	10	6.0	0.5	2.1
5	Acetate	6.3	19.6	24	40	8	13.7	0.55	0.9

NOTES:

S* denotes crystallized sodium carbonate.

In bath 2 the bluestone was precipitated by the carbonate and the resulting sodium sulphate washed out.

Bath 9 contained 36 grams crystallized sodium sulphate per litre.

Bath 11 contained 12 grams crystallized sodium thiosulphate per litre.

Baths 13, 14, 15 were supplied by members of the Electroplaters' Society; the others were made up from receipts found as follows:

1. Watt and Philipp, Electroplating and Refining of Metals, D. Van Nostrand Co., New York (1902), p. 155.
2. Langbein, Electrodeposition of Metals. 5th ed., Philadelphia, 1905, p. 323.
3. Langbein, l. c. 322.
4. Bennett, Trans. Am. Electrochem. Soc., **23**, 237 (1913).
5. Langbein, l. c. 303; Bennett, l. c. 241.
6. Bennett, l. c. 239.
7. Bennett, l. c. 240.
8. Bennett, l. c. 240.
9. Bennett, l. c. 241.
11. Bennett, l. c. 242.

Inspection shows that the numbers under "current density" and those under "free cyanide" do not run parallel; the current density at which a film is formed cannot be predicted from a knowledge of the solubility of silver chloride in the solution. It seemed probable, however, that in every case the formation of a precipi-

tate was due to the same cause. At each square centimeter of the anode surface copper salt is entering the solution at a rate proportional to the current density, and is being carried away into the bulk of the liquid by diffusion and convection; the concentration of copper in the solution at the anode surface will therefore be greater than in the body of the electrolyte, and the difference will increase with increase in the current density. Now, in all the solutions employed, a sufficient increase in the copper concentration causes a precipitate; it therefore seemed probable that solutions in which a small amount of additional copper salt produced a precipitate would give the voltage jump with a low current density, and vice versa.

To test this conclusion, a solution was made up containing 8.00 g. crystallized copper chloride per liter, which was added from a burette to five cubic centimeters of each of the solutions of the table, until a precipitate appeared. In most cases the latter looked blue through the liquid, some, however, were white or yellowish, and one (from No. 11, which contained thiosulphate) was brown; the "end point" of the titration was not very sharp. The number of cubic centimeters of copper solution used is entered under "copper titration" in the table, and comparison of these numbers with those under "current density" shows that the two series run together; by means of a graph it is possible from the burette reading to interpolate very closely the current density at the anode that with any given solution will cause the voltage jump. Not only cyanide, but ammonia or any other substance that holds the copper in solution, can thus be added to the bath to permit the use of a high anodic current density.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 1, 1914, President F. A. Lidbury in the Chair.

THE REPRODUCIBILITY OF THE COPPER ELECTRODE

By Frederick H. Getman.

During the course of the investigations of the electromotive force of various concentration elements which have been in progress in this laboratory for several years our attention has frequently been directed to the variation of the difference of potential between the common metals and solutions of their salts.

While the phenomenon appears to be quite general, in no case has it been more difficult to secure satisfactory reproducibility than with copper electrodes. In fact it is not uncommon to find two copper electrodes prepared in identically the same manner differing by as much as ten millivolts. With a view to eliminating this source of error in our subsequent studies of concentration elements, the copper electrode has been made the subject of a special investigation, some of the results of which are here recorded. While the experimental work was still in progress a paper by Lewis and Lacey[1] on the potential of the copper electrode appeared, in which several of our observations were confirmed and an interesting suggestion as to the cause of the observed irregularities was put forward.

The copper electrodes used in this investigation were prepared in the following manner:

Electrodes I, II, III, and IV, electrolytic copper wires 3 mm. in diameter; electrodes V and VI, platinum plates 4 mm. wide and 15 mm. long, covered with a uniform and adherent electrolytic deposit of copper; electrodes VII and VIII, electrolytic copper wires 1 mm. in diameter; electrodes IX and X, fine platinum loops covered with a deposit of spongy copper obtained by electrolyzing a neutral solution of copper sulphate at high current density; electrodes XI and XII, electrolytic copper wires 1 mm. in diameter embedded in Kahlbaum's copper powder ("reduziert").

Two different types of concentration cell were employed. In

[1] Jour. Am. Chem. Soc., **36**, 804 (1914).

the first, a standard electrode of mercury and mercurous sulphate formed one-half of the element, the other half consisting of the copper electrode immersed in a solution of copper sulphate of known concentration. The following cells of this type were used:

$$Hg\text{–}Hg_2SO_4\text{—}0.01m\ CuSO_4\text{—}Cu \qquad (1)$$

and

$$Hg\text{–}Hg_2SO_4\text{—}0.001m\ CuSO_4\text{—}Cu \qquad (2)$$

The second type of cell consisted of two different concentrations of copper sulphate, each half-element being furnished with one pair of similarly prepared copper electrodes. The following combination was employed:

$$Cu\text{—}0.01m\ CuSO_4\text{—}0.001m\ CuSO_4\text{—}Cu \qquad (3)$$

It is apparent that the electromotive force of (3) should be equal to the difference of the electromotive forces of (1) and (2).

The copper sulphate employed was Kahlbaum's ("zur analyse") and was considered to be sufficiently pure to admit of use without re-crystallization. All of the solutions were prepared by direct weighing, certified volumetric apparatus being used. The standard electrodes were prepared from pure mercury and from mercurous sulphate obtained by the method proposed by Hulett[2]. These substances were triturated in a mortar with the copper sulphate solutions, decanting the clear liquid, and repeating the process in exactly the same manner as in the preparation of a calomel electrode. These electrodes were found to be both constant and reproducible within the limits of experimental error.

The Poggendorff compensation method was used in measuring the electromotive forces of the different cells, an enclosed type of capillary electrometer serving as the nul instrument. All of the cells were placed in an electrically controlled thermostat in which a temperature of 25° ± 0.1 was maintained. In order that thermal equilibrium might be established within the cells, at least thirty minutes was allowed to elapse after placing a cell in the bath before measuring its electromotive force.

The special form of cell which has been in use in this laboratory for several years was employed throughout the investigation. This cell has the advantage that connection between the two

[2] Phys. Rev., 32, 257 (1911).

halves need be established only during the time required to make a measurement of the electromotive force.

The subjoined tables give the results obtained with the different electrodes in the different types of cells; each value of the electromotive force represents the mean of at least three independent measurements.

TABLE I.

$Hg\text{-}Hg_2SO_4\text{-}0.01m\ CuSO_4\text{-}Cu$

Electrode	Electromotive Force	Remarks
I	0.3971	Polished with emery cloth.
II	0.3970	
III	0.3948	
IV	0.3980	
V	0.4107	
VI	0.4107	
VII	0.3985	
VIII	0.3987	
IX	0.4156	
X	0.4155	
XI	0.4223	
XII	0.4213	
I	0.4015	Cleaned by immersing in conc. HNO_3
II	0.3934	

TABLE II.

$Hg\text{-}Hg_2SO_4\text{-}0.001m\ CuSO_4\text{-}Cu$

Electrode	Electromotive Force	Remarks
I	0.3988	Polished with emery cloth.
II	0.3965	
III	0.3906	
IV	0.3972	
V	0.4259	
VI	0.4292	
VII	0.4020	
VIII	0.3932	
IX	0.4404	
X	0.4404	
XI	0.6107	
XII	0.6095	

TABLE III.

$Cu\text{-}0.01m\ CuSO_4\text{-}0.001m\ CuSO_4\text{-}Cu$

Electrodes	Electromotive Force
I, II	0.0143
III, IV	0.0159
V, VI	0.0276
IX, X	0.0275

It will be observed that the variation in the values of the electromotive force in the foregoing tables is greater than the experimental error, while the comparison of the values given in Table III with the differences between the corresponding values given in Tables I and II still further emphasizes the irregular behavior of the copper electrode. Those cells in which spongy copper electrodes were used are exceptional however, the values of the electromotive force being constant and the agreement between the value, 0.0275 volt, given in Table III, and that calculated from Tables I and II (0.4404—0.4155=0.0249 volt), being quite satisfactory.

The electromotive force of the cell

$$\text{Cu} \text{ — } 0.01\text{m } \text{CuSO}_4 \text{ — } 0.001\text{m } \text{CuSO}_4 \text{ — Cu}$$

corresponds to the free energy of dilution of copper sulphate from 0.01m to 0.001m. It may be calculated by means of Nernst's equation,

$$E = \frac{2v}{u + v} \cdot \frac{RT}{nF} \log_e \frac{m_1 \alpha_1}{m_2 \alpha_2}$$

where u and v denote the mobilities of the cation and anion respectively, α_1 and α_2, the degrees of ionization at the molar concentrations m_1 and m_2, n the valence of the cation, F the electrochemical unit and R and T the molecular gas constant and temperature respectively. Taking the values of u and v given by Noyes and Falk[3], ($u_{\text{Cu}^{\cdot\cdot}} = 53.6$ and $v_{\text{SO}_4^{\cdot\cdot}} = 80.0$), and the values of the ionization given by Jones[4] ($\alpha_1 = 0.579$ and $\alpha_2 = 0.870$) we have

$$E = \frac{2 \times 80}{53.6 + 80.0} \times \frac{0.0595}{2} \log_{10} \frac{0.01 \times 0.579}{0.001 \times 0.870}$$

$$E = 0.0293 \text{ volt.}$$

This value is nearly two millivolts greater than that given in Table III, a discrepancy within the limits of experimental error.

To further test the constancy and reproducibility of the spongy copper electrode, a second series of experiments was carried out with freshly prepared solutions and electrodes. In order to

[3] Jour. Am. Chem. Soc., 34, 479 (1912).
[4] Monograph Carnegie Institution of Washington.

ascertain whether the electromotive force of the cell changed with time, readings were taken at frequent intervals during a period of several hours. The results of this second series of measurements in which electrodes IX and X were employed are given in the following tables.

TABLE IV.

$Hg\text{-}Hg_2SO_4\text{-}0.01m\ CuSO_4\text{-}Cu$

Time (Minutes)	Electrode	Electromotive Force
0	IX	0.4140
120	IX	0.4152
180	IX	0.4149
0	X	0.4149
105	X	0.4152
135	X	0.4144
	Mean	0.4148

TABLE V.

$Hg\text{-}Hg_2SO_4\text{-}0.001m\ CuSO_4\text{-}Cu$

Time (Minutes)	Electrode	Electromotive Force
0	IX	0.4502
30	IX	0.4513
150	IX	0.4520
240	IX	0.4532
345	IX	0.4522
0	X	0.4527
60	X	0.4532
	Mean	0.4521

TABLE VI.

$Cu\text{-}0.01m\ CuSO_4\text{-}0.001m\ CuSO_4\text{-}Cu$

Electrodes IX and X.

Time (Minutes)	Electromotive Force
0	0.0301
50	0.0301
135	0.0300
0	0.0333
60	0.0290
135	0.0308
210	0.0275
270	0.0300
330	0.0300
515	0.0283
	Mean 0.0299

The mean value of the electromotive force given in Table VI is only 0.6 millivolt greater than the value calculated by means of the Nernst equation, and it is in fair agreement with the difference between the mean values given in Tables IV and V. It therefore appears that the spongy copper electrode is both constant and reproducible, and the values obtained with it agree closely with the requirements of theory. Lewis and Lacey (*loc. cit.*) reached the same conclusion in their recent investigation of the copper electrode. They also showed that occluded hydrogen plays no part in the electrode process. If this were the case, a change in the concentration of the hydrogen ion would cause a corresponding change in the electrode potential, whereas the addition of 0.001m sulphuric acid to the copper sulphate solution was found to be without effect upon the electromotive force of the cell.

As to the cause of the variations observed with the other forms of copper electrode Lewis and Lacey suggest that these irregularities are to be ascribed to surface strains. When the copper is deposited at sufficiently high current density to cause it to separate out in spongy form, the metal appears to be free from the surface strains characteristic of closely adherent films, and hence is both constant and reproducible. If this hypothesis is correct, it should be possible to produce marked changes in the difference of potential between a copper wire and a solution of copper sulphate by subjecting the wire to such treatment as will bring about a condition of surface strain. In order to test this point an electrolytic copper wire 3 mm. in diameter was introduced into a half-cell containing 0.001m copper sulphate, the other half of the cell consisting of a standard mercury-mercurous sulphate electrode. Before inserting in the cell, the wire was thoroughly polished with clean emery cloth after which it was washed and dried. When the cell had acquired the temperature of the thermostat the electromotive force was measured at frequent intervals for about twenty minutes, the mean of the different determinations being taken as the correct value for the polished electrode. The wire was then removed and subjected to vigorous hammering on an anvil for about five minutes. It was then washed, and after repolishing with emery cloth, was re-inserted in the cell and the electromotive force determined.

The wire was then removed again and heated to redness in the Bunsen flame, after which it was suddenly quenched in cold water. The heating and sudden cooling was repeated twice and then the wire was cleaned by a momentary immersion in concentrated nitric acid, followed by thorough washing in distilled water. The electrode was replaced in the cell and the electromotive force again measured. The results of these experiments are given in the following table.

TABLE VII.

$Hg\text{-}Hg_2SO_4\text{-}0.001m\ CuSO_4\text{-}Cu$

Preliminary Treatment of Electrode.	Electromotive Force.
Polished with emery cloth	0.4330
Hammered for five minutes	0.4339
Heated to redness and quenched	0.4268

It is apparent that hammering has practically no influence upon the electrode potential while heating and subsequent quenching produce a difference of about six millivolts. It is certain that these two modes of treatment must develop surface strains in the electrode and yet the resulting change in electromotive force is negligible. Evidently the strain hypothesis is inadequate to explain the differences observed with the various types of copper electrodes used by us.

A much more satisfactory explanation of the behavior of the different forms of the electrode would seem to be based upon the interesting observations of Cohen and his co-workers[5] upon allotropism among the metals. Cohen and Helderman (*loc. cit.*) have shown from pyknometric and dilatometric measurements that *pure* copper contains several different modifications of the metal, and they further point out that copper is ordinarily in a metastable condition. If we assume, therefore, that spongy copper represents the modification which is stable at ordinary temperatures, the irregularities manifested by the other forms may be traced to their instability under the experimental conditions. This explanation is in harmony with the views advanced by Smits[6] on allotropism. He assumes that each phase of an apparently unary system is composed of at least two molecular species which are in internal equilibrium, and may be looked upon as

[5] Zeit. phys. Chem., **87**, 409, 419, 426, 431 (1914).
[6] Verslag. Akad. Wetenschappen, **22**, 642 (1913).

the components of a pseudo-unary system. A metal which exhibits allotropy must contain several different molecular species, each dissociated to a different extent. Hence when such a metal is immersed in a solution of one of its salts it will yield more than one kind of ion and the nature of the equilibrium will be conditioned by the relative speeds of the different processes.

SUMMARY OF RESULTS.

1. Of the various forms of copper electrode which have been studied, that in which spongy copper is employed is the only form which is constant and reproducible.

2. The irregularities observed with electrodes of massive copper and with electrodes of platinum covered with an adherent deposit of copper, may be ascribed to a condition of metastability. The spongy modification of the metal is to be regarded as the stable modification under ordinary laboratory conditions.

Chemical Laboratory, Bryn Mawr College,
June, 1914.

DISCUSSION.

W. C. MOORE: It seems to me this paper could be made more valuable if several points were made clear about the method of preparing the spongy copper electrodes which seem to be the forms which Dr. Getman could produce at will. First, as to the concentration and temperature of the bath from which the copper was deposited on the platinum loops; second, whether there were any color changes in the deposit as it was being deposited; third, if the deposit showed any crystals; fourth, how he washed the electrodes, and last, how he dried them and kept them from oxidizing.

With reference to the work of Cohen and Helderman[7] they point out that the exact location of the transition point observed by them in the neighborhood of 70° depends on the previous thermal history of the copper, and that there is some reason to believe that a third modification of copper exists. Their results seem to indicate that ordinary copper is not the stable modification at the ordinary temperature; and in this connection it would be

[7] Zeit. f. physik. Chem., 87, 419 (1914).

interesting to have determined the transition point of Dr. Getman's stable spongy copper into the other varieties.

G. A. ROUSH: I disagree with Prof. Getman's conclusions concerning the results given in Table VII. He takes a sample of his raw material, polishes it with an emery cloth, and makes one measurement; hammers it for five minutes, repolishes with emery cloth, and makes another measurement. Then he heats it to redness, quenches it and makes another measurement.

As Dr. Whitney pointed out when he read the paper, in an electrochemical measurement it is the surface material that determines the voltage. It is immaterial what the interior of the material is like; it is the surface that has to be taken into consideration, because it is the surface which comes in contact with the solution. Prof. Getman has taken his first sample and polished it with emery cloth. Any one who is familiar with metallography to any extent, knows that polishing with an abrasive material forms on the surface of a metal a thin layer in which the molecular condition of the material is entirely changed. I should say that is what Prof. Getman had in his first sample. Then he takes the same sample and attempts to put it in a different molecular condition by hammering; but he destroys, to a certain extent, whatever change he did obtain, by polishing with emery cloth, since the second polishing will give approximately the same surface as the sample had after the first polishing. It is only natural under these conditions, to have no material difference between the first and second readings. On the other hand, when he comes to the third test, he takes the sample and anneals it; but instead of polishing it with emery cloth, as before, he dips it into nitric acid and washes it with water, and it is to be expected that a material difference in voltage will result, because in this case the real molecular condition of the metal has not been masked by the thin surface film of polished material.

That any difference at all was obtained in the first and second readings is probably due to the fact that in polishing a sample in this way, it is difficult to distribute the pressure equally over all parts of the surface, and that some parts of the surface might not have been entirely covered with the polished film.

W. R. MOTT: Prof. Burgess and Mr. Hambuechen[8] also

[8] Bulletin of the University of Wisconsin, No. 42. An Experimental Study of the Corrosion of Iron. By Carl Hambuechen, Madison, Wis., 1900. See pages 256, 265, diagrams page 275.

worked on copper under tension as to effect on electromotive force. The results on the observed electromotive force with iron was more than double that calculated from the mechanically stored energy—due in my opinion to a breaking up of an oxide film. The copper (and zinc) gave more consistent results. The diagrams show a remarkable similarity to the stress-elongation curves.

Jos. W. Richards: There is no doubt that when you take a metal and hammer it, as was done in this case, you have changed its molecular tension, and therefore in dissolving it, you might expect to get larger electromotive force from the strained metal.

I think the results here are somewhat consistent, in spite of the fact that the author did polish the material with emery the second time. I notice that after hammering he gets 0.9 millivolt larger electromotive force from the hammered material, and after annealing it, so as to take away the strains, he gets 7 millivolts less. These results are consistent with the fact that straining the metal, as by putting mechanical work upon it, makes it give larger electromotive force, and removing these strains it gives less electromotive force. I think the figures prove it.

The importance of this matter in the corrosion of metals is great, because where a metal is strained or worked, and not worked uniformly, when the metal is carefully examined, we find lines of strain, which are brought out by the application of acids. There are parts of the metal in a greater condition of strain than others. These parts are possessed of a greater potential for solution than the other parts, and when closed in a voltaic circuit, set up with them considerable electrochemical currents. That has been proven pretty well in the case of aluminium, and I think probably in the corrosion of iron and steel, as brought out in some of our recent transactions.

Mr. Getman's result also points to the fact that when you are trying to get a scientifically pure material, absolutely reproducible, you must try to secure metal which has no physical history. If you take a metal which has a physical or thermal history, you will get varying results if your tests are delicate enough. In order to get reproducible results you must use the metal in such condition that its past history has the minimum effect upon the experiments.

F. C. FRARY: It would be very interesting if Mr. Getman would test some of his spongy copper for sulphide. It is generally found in electro-analysis that copper deposited in a spongy state weighs more than it should, and is dark in color. Both these facts are ascribed to the presence of sulphide. It is also important to check the neutrality of the solution made up from supposedly C. P. copper sulphate. A trace of acid would cause varying results to be obtained as the acid gradually was used up by the electrodes.

W. LASH MILLER: The influence of the surface is readily seen by looking at the deposit on a burnished copper cathode; if the current flows for a short time only, the deposit is not uniform, but follows otherwise invisible lines made by the burnishing tool.

JOS. W. RICHARDS: It may be possible that when the copper is deposited in the spongy state, with a high current density, it contains a small amount of hydrogen. If it happens always to have a uniform amount of hydrogen, then it is all right, and the electrode might be reproduced within the limits of probable experimental work. I think if this copper were examined carefully it would be found to contain a certain amount of hydrogen.

W. C. MOORE: With reference to the point Prof. Richards just brought out, this work of Cohen and Helderman refers to another article in which mention is made of the fact that spongy copper sometimes turns dark in precipitation, and this dark color is possibly due to an alloy of hydrogen and copper. I asked, for that reason, whether any color changes were observed. These authors mentioned by Cohen and Helderman, referring to the phenomena observed on electrolyzing copper solutions with a high current density, point to the fact that no hydrogen is evolved from the dark deposits of copper, while it is evolved from the bright deposits of copper; and I have recently observed when the darker deposits become light, that hydrogen is evolved from them *then,* and not until then do they become light.

H. C. CHAPIN: Suppose these dark spots were copper oxides? Would not that react on the hydrogen, and prevent its evolution?

JOS. W. RICHARDS: I do not think there is likely to be copper oxide deposited on the cathode. If you electrolyze a solution of dilute copper sulphate, you get at first a bright deposit of copper.

But you can keep on electrolyzing until you get hydrogen; and as hydrogen commences to appear, the copper commences to get spongy, so there seems to be a direct connection between the spongy condition of copper and the evolution of hydrogen.

W. C. Moore: The work I did was with a solution which was acid, and there was little chance for copper oxide to form. Hydrogen was evolved from this black copper and then it turned light; it was black at first and light later; whenever the copper deposited at first was *bright* it allowed hydrogen to be evolved therefrom *immediately*.

Frederick H. Getman (*Communicated*): The spongy copper electrodes were prepared by electrolyzing, at room temperature, a solution of copper sulphate containing approximately 375 grams per liter. The current density was about 2.1 amperes per square centimeter of electrode surface. The copper separated in an amorphous form, the deposit having a reddish-brown color. The platinum loop covered with the deposit of copper was washed successively in distilled water, alcohol, and ether, after which it was immersed in the solution of copper sulphate in the experimental cell.

It has been shown by Lewis and Lacey (*loc. cit.*) that oxidation plays no important rôle in the behavior of copper electrodes prepared in the foregoing manner. Identical results were obtained with the electrodes when special precautions were taken to insure the compete absence of oxygen. The same authors also proved that occluded hydrogen plays no part in the electrode process by carrying out a series of experiments in copper sulphate solutions containing 0.001 N. sulphuric acid. If hydrogen were occluded in the electrodes the addition of $H^{\cdot}$ ions to the electrolyte would cause a variation in the potential. No change, however, was observed.

The determination of the transition point of the spongy copper is a phase of the problem which I hope to report upon in the future.

No tests were applied to ascertain whether the electrodes contained traces of copper sulphide.

As to the irregularities observed with stick electrodes, it is evident that further experimentation is necessary before their anomalous behavior can be satisfactorily explained.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 1, 1914, President F. A. Lidbury in the Chair.

THE ELECTROLYTIC DETERMINATION OF NICKEL.

By W. Judson Marsh.

Among the methods proposed for the electrolytic determination of nickel, that of A. Classen, who uses a solution containing an excess of ammonium oxalate, is quite generally recommended by the standard text books on quantitative analysis. The method is also mentioned favorably in some of the books on electro-analysis. Work in this laboratory had already shown that this method was not especially accurate or easy to carry cut, and so it was decided to repeat certain work already carried out and to test out certain other suggestions made by Professor Bancroft of this laboratory.

Much work has already been done on the electrolytic determination of nickel and its congener cobalt, but a complete bibliography of such research is not required in the present article[1].

In 1902 Akerberg[2] made a study of the rapidity of the electrolytic decomposition of oxalic acid in the presence of sulphuric acid, in which he came to the following conclusions:

1. In H_2SO_4 solution oxalic acid is but little decomposed by the electric current.

2. Platinized platinum anodes cause the decomposition of oxalic acid, *i. e.*, it is oxidized at the anode.

3. Reaction is monomolecular, *i. e.*,

$$H_2C_2O_4 + O = H_2O + 2CO_2$$

In 1909, G. Lambris[3] reported a further research on "The Taking-up of Carbon by Metals in the Case of Electrolysis from Aqueous Solution, with especial reference to Nickel." In this article the author reviews previous work on the electrolytic deposition of carbon and describes his own experiments minutely. It had been found that certain carbonaceous material was deposited along with nickel in the method suggested by Classen

[1] Zeit. anal. Chem., **19**, 314 (1880).
[2] Zeit. anorg. Chem., **31**, 161 (1902).
[3] Zeit. Elektrochemie, **15**, 973 (1909).

and the author attacked the problem of explaining and preventing this. He came to the following conclusions:

1. The absorption of carbon by the nickel is entirely due to a gas reaction.

2. The gases which introduce the carbon are CO,CO_2 and C_2H_2. Such gases as are reduced beyond C_2H_2 do not introduce any carbon.

3. Oxalic acid is partly reduced to C_2H_2 upon platinum and nickel, but not when Cu, Fe, or Sn cathodes are used. Therefore C_2H_2 is only a carrier of carbon to metals (cathodes) in electrolysis.

4. The carbon in electrolytic Ni is present in the form of carbide.

5. A porous porcelain cup placed about the cathode prevents the deposition of carbon with the nickel.

6. The introduction of $(NH_4)_2CO_3$, C_2H_2, CO_2 and CO into the cathode compartment causes the separation of carbon along with nickel.

7. When C_2H_2 is used, the carbon content is very nearly the same as in the case of the oxalate electrolysis.

Lambris also did much other work on the nature of this deposit of carbonaceous matter, and came to the conclusion that it existed as a carbide, which when separated out for analysis invariably yielded heavy hydrocarbons, light hydrocarbons, carbon dioxide, and hydrogen on treatment with dilute HCl. He also concluded that certain small amounts of ammonium salts were held (adsorbed) by the carbon itself, which separates on dissolving the metal deposit. He was able to obtain results from 2 to 2.5 percent in excess of the weight of nickel known to be present (*e. g.* 8.6 mg. carbon in a deposit of 0.4403 g. nickel).

Classen's method is described in his book, "Quantitative Analysis by Electrolysis." (1903 edition, p. 160.) "Metal present as sulphate.

Substance added: 6 to 8 g. ammonium oxalate.

Total volume of solution: 120 cc.

Temperature: 60° to 70°.

Current density at cathode: $N.D._{100} = 1.0$ ampere.

Potential difference: 3 to 4 volts.

Time required: about 3 hours."

Classen advises the use of ammonium sulphide to test the solution for the last traces of nickel. His book gives short descriptions of the methods of Fresenius and Bergmann [$(NH_4)_2SO_4$ + free NH_4OH in solution], Oettel (chloride solutions), Smith (cyanide solution), Fernberger and Smith (phosphate solution), Campbell and Andrews (phosphate and excess NH_4OH solution) and Gooch and Medway [$(NH_4)_2SO_4$ + NH_4OH with rapid rotation]. He gives no further data as to the conditions or results of his method for nickel; but under his oxalate method for tin, which is quite similar, he claims that the method is sufficiently accurate to yield results suitable for the atomic weight determination of tin (page 212).

Attempts have been made in this laboratory to use this oxalate method for the determination of nickel, cobalt, and tin, but only in the last-mentioned case (tin) has it proved suitable for accurate electroanalysis. Briefly, some of the disadvantages or inconveniences of the method are: the use of special and expensive apparatus, the necessity of plating the platinum dish cathode with copper or silver, the long and uncertain time required for a determination (3 to 7 hours), extra trouble of heating the solution to a constant temperature, evaporation of the electrolyte and the consequent tendency of salts to "crawl" out on the stem of the anode and upper edges of the dish, the separation of the nickel as peroxide on the anode toward the end of the run, the necessity of siphoning out the solution and adding distilled water at the end of the run and the uncertainty as to results obtained, these usually running high due to the co-deposition of carbonaceous material.

In the description of the method no mention is made of this last and most troublesome tendency, and, of course, no suggestion as to any means of obviating it. With this in mind the present research was begun with the following objects in view:

1. Repetition of certain parts of the work of Akerberg and Lambris.

2. To find the conditions which give the worst results, *i. e.*, the highest percent of deposited carbon, etc.

3. To find the conditions which give the best results by this method.

4. If possible, to modify the method so as to render it more accurate and rapid.

5. To test out the effect of certain addition substances.

6. If not possible to improve the method sufficiently, to work out and give in detail some simpler, more rapid, and more accurate method.

Very little need be said as to the repetitions of work by Akerberg. From the statement of the former to the effect that oxalic acid was but little decomposed by the current when sulphuric acid was present, it was thought that possibly the presence of the sulphate ion might prevent the break-up and consequent oxidation of the oxalate ion and consequently do away with the deposition of carbon at the cathode, the theory being that the oxalic acid is oxidized at the anode to $2CO_2$ and H_2O, and that the CO_2 then diffuses, in part, to the cathode, where it is reduced and some acetylene is formed, which then may react with the freshly deposited nickel to form a carbide of nickel. The use of H_2SO_4 in this case is not permissible and so $(NH_4)_2SO_4$ was tried, without the desired result. A solution, the Ni content of which had been previously determined by the excellent dimethylglyoxime method of Brunck[4], was found to contain 0.1698 g. Ni per 50 cc. solution. Using the apparatus and conditions recommended by Classen with additions of 3, 5, and 10 grams $(NH_4)_2SO_4$, deposits were obtained weighing 0.1730 g., 0.1748 g., 0.1719 g. In every case 50 cc. samples were taken carefully from a reservoir—burette bottle (at or very near 20° C).

An attempt was next made to repeat the work in which Lambris obtained the highest amounts of carbon along with the nickel deposit—the amount being judged by the difference in weight of the deposit and the weight of nickel in the solution used (*i. e.*, 0.1698 g. Ni per 50 cc. solution). Unsuccessful attempts were made to cause the nickel of the deposit to dissolve in $KCl.CuCl_2.2H_2O$ solution as used in the determination of total carbon in iron or steel. It was found that hydrochloric acid of a concentration sufficient to dissolve the deposit left little or no solid residue of carbonaceous material, which is in accord with the findings of Lambris. It was found possible, however, to

[4] Zeit. angew. Chemie, 1844 (1907).

obtain some of this carbonaceous material by adding distilled water and then nitric acid drop-wise until the deposit began to dissolve very slowly as shown by the evolution of small gas bubbles from the surface of the deposit. The reaction, however, tended to speed up as heat was evolved and more distilled water was added. With care it was found possible to obtain from ½ to ⅔ of the weight in excess of the true weight of nickel. The amount of this solid residue, however, was necessarily small (a few milligrams) and, when analyzed by the oxygen combustion method, the results were not very satisfactory, indicating that the product was from 77.5 to 91 percent carbon. This was not considered surprising, however, as the material seemed to vary considerably in color, size of particles, and physical form. Usually it was nearly black, but on two occasions it appeared distinctly brownish. Usually it was finely divided and granular in appearance, but sometimes it was flocculent and filmy. It seemed to cling tenaciously to small amounts of ammonium salts. Lambris reported that he was unable to notice any carbon when Cu, Fe, or Sn cathodes were used. In this laboratory very considerable amounts of carbon have been found when a solution of copper carbonate is dissolved in a slight excess of acetic acid and the solution electrolyzed, using a potential difference of 2.5 to 3 volts and a current density of $N.D._{100} = 1$ to 2 amperes in a total volume of 150 cc. Also in Classen's oxalate method for tin, numerous high results have been obtained. A tin solution made from Kahlbaum's C. P. Tin so that 50 cc. was exactly equal to 0.3000 g. Sn gave the following results:

Series I	Series II
0.3008	0.3036
0.3010	0.3040
0.3007	0.3041

The results in the second series were higher than those in Series I because they were run from 15 minutes to one-half hour longer than those in Series I. Conditions used were 4 g. $(NH_4)_2C_2O_4$, 8 g. $H_2C_2O_4$ at 1 ampere per 100 sq. cm. and 3 volts, using a rotating copper gauze cathode (see below). In spite of Classen's note to the contrary, this method does not seem well suited to the determination of the atomic weight of tin. If the electrolysis was allowed to run for one hour, the results

were fairly good, but they seemed to go much too high when allowed to run longer.

In the introductory part of his book Classen recognized several phenomena of a nature apparently similar to the above cases in which the oxalate radical seems to furnish a source of the carbon deposited with metals. For example (on page 39 of his book) he noted that the carbon dioxide was formed by the oxidation of the acetic acid radical, which then might also yield ethane and even ethylene. Octane, isobutylene, ethylene, carbon monoxide, formaldehyde, formic acid, etc.—he said were also formed on the break-up of various organic acids.

The attempt to find conditions which yield the highest amounts of deposited carbon gave no new results. The percentages of carbon deposited, as indicated by excess weight of the deposit, were of much the same magnitude as those obtained by Lambris, *i. e.*; about 2.5 percent.

Next, numerous runs were made in an endeavor to improve the method, but without much success. When the temperature is kept close to 50° and water is added from time to time to replace that lost by evaporation, the method gives fair results if allowed to run 2.5 to 3 hours. These results are usually a little low, however, as some nickel remains in the solution. This amount is too small to be shown by the $(NH_4)_2S$ test, but shows up readily on using dimethylglyoxime as a spot-test reagent. This indicator is now used in this laboratory as an end test for both nickel and cobalt, giving a reddish-pink coloration with Co and a pink precipitate with Ni (in presence of NH_4OH or NaAc). Variation of the amount of $(NH_4)_2C_2O_4$ had but little effect as long as the amount present exceeded 5 grams per 120 cc. of solution containing *ca.* 0.17 g. Ni. The addition of 1–2 gram of urea had a beneficial effect. The tendency for NiO_2 to separate at the anode toward the end of the run was eliminated by addition of a little NH_4OH or "glycerol mixture."[5] or by decreasing the current and voltage for a few minutes near the end of the run.

Since none of the above changes improved the method sufficiently to render it as rapid and accurate as a good electrolytic method should be, it was decided to try the effect of certain

[5] E. F. Smith: Electro-Analysis.

addition substances (known to electroplaters as "dopes"). It was thought that some reducing agent might be found which would react with the oxygen formed at the anode and by so doing decrease the time required and the amount of carbon dioxide formed and consequently the amount of carbon deposited. Those tried were hydrazine sulphate, hydroxylamine sulphate, sodium nitrite and sodium sulphite. Of the first three hydrazine sulphate was the only one to produce a noticeable effect, when it was added to the extent of 5 grams it produced a blackening of the deposit, possibly due to the formation of nickel sulphide.

This last seems more probable in the light of the unexpected behavior of the last reducing agent, sodium sulphite. Although this has not been sufficiently investigated, it is certain that its effect is surprising and for this reason it is to be studied more thoroughly. Briefly the phenomena noticed were as follows: On addition of 5 grams of Na_2SO_3 (Baker's C. P.), all other conditions being those employed by Classen, a black flocculent precipitate begins to form almost as soon as the circuit is closed. This precipitate increases rapidly in amount and the odor of hydrogen sulphide is perceived. Thinking that this phenomenon might be due to some chemical reaction, a blank was made which showed this not to be true. Other blanks were run, the first showing no such behavior when the 6 to 8 grams $(NH_4)_2C_2O_4$ was electrolyzed with 5 grams Na_2SO_3 in 120 cc. of water at 50°C., and the second also gave a negative result when the nickel solution and 5 g. Na_2SO_3 was electrolyzed, all other conditions being the same except that no $(NH_4)_2C_2O_4$ was present.

At first it was thought that this precipitate might be some carbonaceous material such as that which deposited with the nickel, but on filtering it off and washing it, the material dissolved quite easily in warm 6NHCl with evolution of H_2S and the formation of the characteristic green solution of nickel chloride. It was evident that 120 cc. of a nickel sulphate solution is quickly reduced to black NiS in the presence of 6 to 8 g. $(NH_4)_2C_2O_4$ and 5 g. Na_2SO_3.

Then, on washing out the Classen dish in which this run had been started, it was noticed that a firm deposit had been formed

on the copper with which the dish had been previously plated. This deposit appeared much like the ordinary good nickel deposit, but was somewhat darker in color. Previously certain very thin films of iridescent metallic sheen had been noted floating off from the dish cathode when the solution was rapidly stirred. These films were very similar to those often noticed when H_2S is passed into solutions for the precipitation of metals in qualitative analysis, especially in case of lead solutions. Although this work has not yet been confirmed it seems reasonably certain that these films as well as the firm, even, metallic deposit on the copper were both nickel sulphide. It may be possible that in dissolving the deposit with 1 : 1 HNO_3 some error of manipulation was made, but it is certain that after washing the deposit carefully, a considerable quantity of hydrogen sulphide was given off when the nitric acid was diluted. This might seem to indicate that a form of nickel sulphide not unlike the mineral millerite (sulphide of Ni) had been formed. This mineral[6] is said to have a "grayish iridescent tarnish" and it is claimed to have been prepared artificially.[7]

The addition of small amounts of urea (1 to 2 g.) seemed to cut down the time required for the completion of a determination, but no effect on the carbon deposition was noted.

Finally it seemed possible that a decrease in the size of the anode might tend to lessen the oxidizing effect at that electrode, as the oxygen would be less liable to remain in the solution. When, however, a platinum wire was used as anode, it was found that the consequent increase in anode density augmented the tendency of nickel to deposit as peroxide on the anode. For this reason it was necessary to abandon this idea.

At this point it was thought best to test out some other method along with a simpler form of apparatus. It was hoped to adopt and modify some of the successful methods of the electroplating industry so as to work out a procedure satisfactory to the analyst. Thinking that such a method would be more liable to yield the desired results if the solution should be as nearly neutral and of as unchanging composition as possible, a number of plating methods were tried out, including a boric acid solution, the same

[6] Dana's Mineralogy, p. 70-71.

[7] Weinschenk: Zeit. Kryst., **17**, 500 (1890); Baubigny: Syn. Min., **4**, 168 (1817).

with various addition substances, a solution containing an excess of borax, a solution containing an excess of sodium pyro-phosphate and ammonia, a solution containing a slight amount of formic acid and an excess of sodium formate, a solution containing a small amount of free lactic acid and an excess of lactate (NH_4 lactate), a solution containing free phosphoric acid and NaH_2PO_4 and finally the well-known ammonium sulphate-ammonia solution.[8]

Of all the solutions above mentioned, the last was the most satisfactory. Some would not run to completion, some gave good deposits at first but unsuitable for analytical purposes due to "burning," etc., at the end, most of them were very slow, etc. After considerable work on this method the following very satisfactory solution was found:

Nickel present (about 0.2000 g.) as $(NH_4)_2SO_4.NiSO_4.6H_2O$.
Ammonium hydroxide 15–20 cc. (Sp. g. 0.96).
Ammonium sulphate 5 grams.
Amperage 0.8 ± 0.1.
Potential difference 3.5 ± 0.2 volts.
Time required about 25 minutes. Room temperature.
Nitric acid, nitrates, and chlorides must not be present. Total volume of solution made up with H_2O to 200 cc.

Electrolyze in a 350 cc. beaker using a 10 g. platinum wire gauze flag anode (52 mesh and 6 x 4.5 cm.) and a rotating (550–650 R.P.M.) pure Cu gauze (50 mesh and 5 cm. high x 3 cm. diameter) cathode. This electrode combination is much less expensive than the Classen dish and disk of platinum, requiring only one-fifth as much platinum as the latter. The Cu wire gauze cathode can be made easily, quickly, and cheaply. Stiff, twisted Cu wire is used for the stems and small Cu tacks are used to rivet the gauze together. One such cathode can be used for a great many determinations, as a fresh surface for deposition may be obtained by dipping a used cathode in 1 : 1 HNO_3 for a few moments. When well made and properly handled these cathodes which cost but a few cents and a few minutes work give results exactly as good as any platinum cathode. This makes it possible to run many more determinations than is usually possible with

[8] E. F. Smith: Electro-Analysis, pp. 122-130.

more expensive forms of apparatus. These cathodes have also been used successfully in the laboratory for the determination of copper, tin, cobalt, cadmium, bismuth, silver, and zinc. They have also been used in combination with the platinum wire gauze anodes for the determination of lead and manganese, both of which are deposited on the anode as peroxides.

The accompanying table of results obtained by eleven different students using this method will give some idea as to the value of the method to those not especially skilled in quantitative manipulations.

ELECTROLYTIC DETERMINATION OF NICKEL.

Solution = $NiSO_4.(NH_4)_2SO_4.6H_2O$ (*ca* 21 g. per liter)

Weight of nickel in each run[9] = 0.1336 g. + 15 cc. NH_4OH, sp. gr. 0.96; diluted to 200 cc.

50 cc. nickel solution. Potential difference 3.5 0.2 volts.

Current	Time	Weight of Ni found	Error of Method
0.7 amp.	20 min.	0.1359 g.	+0.0023 g.
0.7 "	20 "	0.1337	—0.0001
0.7 "	20 "	0.1296	—0.0040
0.7 "	20 "	0.1340	+0.0004
0.7 "	20 "	0.1310	—0.0026
0.8 "	30 "	0.1348	+0.0012
0.8 "	30 "	0.1338	+0.0002
0.8 "	30 "	0.1346	+0.0010
0.8 "	25 "	0.1334	—0.0002
0.8 "	25 "	0.1334	—0.0002
0.8 "	25 "	0.1332	—0.0004
0.8 "	25 "	0.1338	+0.0002
0.7 "	25 "	0.1341	+0.0005
0.7 "	25 "	0.1336	0.0000
0.7 "	25 "	0.1340	+0.0004
0.7 "	25 "	0.1336	0.0000
0.7 "	25 "	0.1332	—0.0004
0.7 "	25 "	0.1344	+0.0008
0.8 "	25 "	0.1339	+0.0003
0.8 "	25 "	0.1339	+0.0003
0.8 "	30 "	0.1344	+0.0008
0.8 "	30 "	0.1331	—0.0005
Average..0.75 "	25 "	0.1336 g.	+0.00002 g.

[9] The amount of nickel per 50 cc. of solution (0.1336) was determined by averaging a series of fine determinations of Ni by the dimethyl glyoxime method.

The writer wishes to acknowledge and express his gratitude for the many timely suggestions of Professor Bancroft. Much valuable assistance was also rendered by Dr. G. E. F. Lundell and Mr. A. M. Hart.

Cornell University,
June, 1912.

DISCUSSION.

F. C. FRARY: The method recommended is a very slight modification of that of Fresenius and Bergmann, to which Dr. Marsh refers, and I can corroborate his statement as to the accuracy and satisfactory character of the determination, as we have used it at the University of Minnesota for a number of years, We find, however, that it is not necessary to rotate the electrode. With most metals the rotation of the electrode or electrolyte is necessary if higher currents are to be used. We prefer a nickel cathode to a copper one, as there is less error due to oxidation at the point where the electrode is at the surface of the solution. Several coats of nickel can be deposited, one on top of the other, until the meshes of the gauze are nearly filled, when part of the nickel is removed with acid, and the electrode is ready for use again.

W. JUDSON MARSH: We also used nickel gauze. I am not familiar with the method of using the cathode without rotation. I am glad to know of that. We had noticed that there was some oxidation effect at the stem of the copper electrode, but it was very slight. I think with care and occasional washing that these accumulations can be cut down very much. The nickel gauze electrodes, however, cost more than the copper.

JOS. W. RICHARDS: In industrial plating there is a different thing aimed at from what you have in electro-analysis. In industrial practice you are using nickel anodes and not trying at all to extract the nickel from the solution. In electro-analysis you are trying to take the last trace of nickel out of the solution; the two are different problems, entirely different in their nature,

so that it is hardly likely that the industrial method would necessarily be successful for the analytical method; on the other hand, the analytical method will very frequently give information of use to the industry.

W. Judson Marsh (*Communicated*): In reference to continued use of the copper wire gauze cathodes, I would call Dr. Frary's attention to the statement at the bottom of page 87 of my paper. It was not at all uncommon to use these cathodes for 35 or 40 determinations each. If one should become weakened it could be strengthened by causing either copper or nickel to be plated on the weakened part.

In reply to Professor Richards' statement that the practice in industrial plating and electro-analysis are quite different, I would say that our object was to make such simple modifications of the methods of the electroplating industry that it would be possible to use them as methods for control of the plating baths, etc.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 1, 1914, President F. A. Lidbury in the Chair.

ON ELECTROMETRIC TITRATIONS.

By Henry Ziegel.

The following concerns a possible technical application of the change in potential at the end point when a bichromate solution is reduced by a solution of ferrous salt.

The change in potential in a solution at the end point of a titration has been studied by several workers during the last fifteen years.[1] Recently Hildebrand,[2] and especially Forbes and Bartlett,[3] have studied this change in potential in the ferrous iron-bichromate titration. Under the conditions observed by the latter authors, this potential change is about 0.2 volt and even greater. The author has recently been successful in applying this change in potential to the regulation of the burette stopcock in such a manner that the stopcock closes automatically when the end point of the titration is reached.

The apparatus used was similar to that of Forbes and Bartlett,[3] with the following changes. In place of the usual galvanometer, a Weston No. 30 relay was used. Thus when the change in potential took place, immediate contact was made in a circuit having in it a telegraph sounder with a narrowed arm seven inches in length from fulcrum to tip. This sounder arm tip was connected to one side of the burette stopcock tip by means of a piece of fine chain. Final connection was made to the stopcock tip by a small brass cap cemented on with sealing wax. Such a device closed the stopcock, and a similar sounder was adjusted to the other extremity of the stopcock so that the latter would be opened by it. Thus, at the initial potential in the solution to be titrated, the arm of the Weston instrument completed a circuit which opened the burette stopcock, and at the end point

[1] Ostwald, Luther, 3d ed., p. 453.
[2] J. Am. Chem. Soc., Vol. 35, p. 846.
[3] Ibid, 35, p. 1527.

the change in potential caused the needle to swing a few hundredths of an inch, open the above circuit, and close the circuit containing the sounder which caused the stopcock to close. That is, the change in potential at the end point can be caused to close the burette stopcock mechanically, thus showing the first step in the accomplishment of titrations by other than manual operations.

The rapid stirring necessary was accomplished by using the elongated shaft of a cheap battery motor. This shaft was heavily platinum plated, and tipped with a deeply nicked disc of sheet platinum. This platinum formed the oxygen electrode. An ordinary form of calomel electrode was used. This latter, the wire bridge and burette used deserve no further comment. The galvanometer used was a rewound stock Weston No. 30 relay.

The procedure was to set the beaker containing the bichromate in place, close the stirring motor circuit, and then close the switch controlling the two sounders. The burette would then deliver until the end point was reached, when, due to the potential change, the stopcock would be closed by the second sounder. These two sounders were actuated by the delicate Weston instrument mentioned above.

Conditions: 20.00 cc. $K_2Cr_2O_7$ + 10 cc. H_2SO_4, Sp. G. 1.84, + about 125 cc. H_2O in beaker. $FeSO_4$ sol. in burette. 1 cc. of each sol. = about 0.01 g. Fe.

Data.

Cc. Fe^2 sol. added automatically.	
19.68	19.62
19.50	19.71
19.71	19.59
19.79	19.55
19.70	19.64
19.40	19.66
19.67	19.66

Spot plate check, done manually.

Ferrous Solution taken	Bichromate Solution added
19.50	19.84
19.50	19.80
20.00	20.39

In short, solutions of Cr^6, or solutions of Fe^2 to which known excess of Cr^6 had been added, may be titrated entirely mechanically, the only manual movements being the throwing of one or two switches.

The author proposes to apply this apparatus to the automatic titration of iron after the direct procedure of Hildebrand (*l. c.*) and to the titration of zinc by ferrocyanid.

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DISCUSSION.

G. A. ROUSH: In the last number of the Journal of the American Chemical Society, Vol. **36**, No. 9, is given a report of Van Suchtellen and Itano, who have carried on still further experiments in this same sort of work. They have not gone so far in the matter as to make the titrations automatic, but they have extended the knowledge of the voltage changes in titrations to various other combinations.

From the combined results of Ziegel with these other two men, it begins to look as though it may be possible to cut down the amount of work involved in the routine analytical work in such places as iron and steel laboratories.

WARREN F. BLEECKER: The actual labor in titrating is about negligible compared with the labor in preparing the sample and getting ready for titration. Any saving you would make in time and labor would be more than offset by possible inaccuracies in the reading.

Something about as serious as this, which I developed about twenty years back, is titrating a solution of permanganate and passing a ray of light through a selenium cell. These things seem to be more or less academic, and I do not believe you will find a widespread field for them in commercial laboratories.

F. A. LIDBURY: I entirely disagree with Mr. Bleecker's suggestion that this kind of work is of little practical importance. It is necessary to realize, of course, that no one wants to substitute electrometric titration for ordinary titration, in cases where the latter is a perfectly satisfactory procedure, but there are certain classes of work in which electrometric titration has con-

siderable advantages over titration with colored indicators, and there are some cases in which it is the only thing that can be used with satisfactory results.

For instance, in the titration of small amounts of chloride in water it is extremely difficult to get any kind of satisfactory results with the chromate indicator, but it is very easy to get an accurate, sharp endpoint by using a silver electrode as an indicator.

There is this peculiarity about electrometric titration, that the curve of voltage against a number of cc. used rises (or falls) with increasing rapidity as the endpoint is reached. In the neighborhood of the endpoint the change in voltage for each drop added becomes very considerable. This is the reason for the extreme sensitiveness of the electrometer as an indicator. At the same time it is rather difficult to carry out the titration manually, as you do not have the colored warning of the approaching endpoint and it is consequently very easy to over-shoot. The mechanical methods of stopping the titration at a predetermined voltage endpoint, which is described in this paper, gets rid of that difficulty entirely, and seems to me to form a distinct step forward in the ease of application of electrometric titration.

W. R. Whitney: If you add the measurement of the electrical resistance to this scheme of measuring electromotive force, you have quite a group of possible electrical titrations. Take acid and alkali titrations, for example, since your acid represents your highest, and the neutral salt the lowest, in many cases when you have acid or alkali in a solution which is highly colored or carries much suspended matter, which prevents using ordinary indicators, then the electrical resistance changes may be used as a means of determining the neutral point in the tiration. As you add alkali, the resistance of the solution falls off, and when it passes the neutral point, it begins to increase.

W. Lash Miller: I think Mr. Ziegel has chosen a very good instance for the application of his method; especially since the analytical procedure he proposes to replace is one of the slow and troublesome volumetric analyses with external indicators or "dab analyses." From the data, moreover, it appears that Mr. Ziegel obtained much more concordant results with his machine than he did when working in the usual way.

JOS. W. RICHARDS: I would like to criticize Mr. Bleecker's statement that because the analysis consists of a number of different parts, it is not worth while to bother much about improving the titration. If you apply that principle to the titration, you could apply it to any proposed improvement and diminish its probable value. In a large laboratory there is usually considerable division of labor. If you can increase the accuracy of the titration process and decrease the time required, you are directly decreasing at least one man's work in that proportion.

WARREN F. BLEEKER: I am willing to admit that the method is accurate and it is perfectly feasible to use it; but I think the objection to it is the fundamental prejudice against a complicated electrical device in the laboratory, where you cannot afford expert attention, and most of our laboratories are conducted on that basis.

WILLIAM BRADY: In regard to the "spot" titration, I would say in general that it is not in use. We use the permanganate, and endpoint is determined by the flash, and very quickly. It seems to me that this process for the titration of iron in steel laboratories is not applicable. We looked it over, and thought it might be a good thing, but finally decided it is not equal to the permanganate titration in regard to speed.

G. K. HERZOG: Did I understand you to say that this method was particularly good for small amounts of chlorides?

F. A. LIDBURY: Yes, you use in that case a silver electrode and titrate against silver solution.

G. K. HERZOG: The St. Lawrence River Power Co., of Massena, N. Y., recently tested a number of large turbines by the chemical method as described by Mr. B. F. Groat,[4] under whose direction the work was done. It is a well known fact that current meters are very unreliable for measuring the flow of a turbulent stream such as the tail-race of a large turbine.

The chemical method is much more accurate for this purpose. It consists essentially of dosing the water at some point above where it enters the turbine at a certain definite rate with a solution containing a known number of grams of some reagent per liter and then determining the number of grams per liter of

[4] Pitot Tube Formulas, *Proc. Eng. Soc. West. Pa.*, **30**, 366-380 (May, 1914).

the reagent in the tail-race water. It is evident that from these values the rate of flow can be determined.

In the tests carried out at Massena, the reagent used was sodium chloride, over 400 tons of this being used. The amount of salt in solution was determined by titrating with centi-normal silver nitrate solution, using potassium chromate as an indicator. Approximately 4,000 samples of one-half liter each were taken. These were all evaporated down to 10 cc., necessitating the use of three shifts of chemists working eight hours per shift with two men per shift. The accuracy obtained was within one-tenth of one percent. It is quite evident that if electrometric titration will give results of equal accuracy without the necessity of such an enormous amount of evaporation, it could be used to advantage in work of this kind.

F. C. Frary: It appears from the paper that the difference between the machine and the manual titration is rather large; twenty c.c. of the same bichromate solution requiring respectively 19.6 and 20.4 c.c. of ferrous sulphate, a difference of 0.8 of a c.c. It would appear that the machine shuts off the ferrous sulphate too soon.

H. Ziegel: I made three spot-plate checks which agreed within a few tenths of one percent of the electrometric automatic titration.

G. A. Roush: I will admit what Mr. Brady says is true in most cases, but there are still laboratories which use the bichromate titration, although not many. Not long ago I tried to get a blast furnace laboratory to substitute the permanganate for the bichromate, but they refused to do so.

In regard to Professor Frary's objection, since this machine can be set to shut off the titration at any desired voltage you should be able to adjust the operation to check exactly with the manual titration.

H. Ziegel (*Communicated*): The author wishes to thank Professor Forbes for his kind interest evinced in this work. He is not acquainted with the work of Van Suchtellen and Itano.

So much pressure was brought to bear on the author that he at one time felt with one of the speakers that any saving in time and labor would be more than offset by possible inaccuracies in

the reading. (Actually three spot-plate checks instead of six were made and these checks agreed within a few parts per thousand of the mean of the electrometric automatic titration). Accuracy of 0.2 c.c. is easily attainable even with the crude facilities at the author's command, and several subsequent titrations have been less than 0.1 c.c. apart; that is, any criticism of the actual facts of this titration even when automatic, as a possible improvement would be in the nature of a chimera.

The author has made a few experiments using bichromate in the burette and ferrous solution in the beaker. In this case the end point is harder to get, due to lack of the "anomalous rise," discovered by Forbes and Bartlett, which gives rise at the end-point to a comparatively large change in potential.

The author has completed his work on this phase and anticipates further applying the hydrogen electrode by causing current changes in a solution being titrated to record changes in voltage on a moving sheet of paper in such a way that the number of c.c. of solution required for a titration will be recorded permanently on a chart.

Acknowledging the assistance derived from the work of other investigators the author hopes he has made or indicated to some chemists or superintendents a possible improvement in ordinarily known chemical analysis.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 2, 1914, President F. A. Lidbury in the Chair.

ELECTRODEPOSITION OF LEAD FROM LEAD ACETATE SOLUTIONS

By Frank C. Mathers.

Lead fluosilicate and lead perchlorate baths are the only ones that have been used successfully in lead plating. The great disadvantage or objection to them is that they must be made by special apparatus and by troublesome methods. The actual electroplating with either of them is very easy.

The deposits of lead which were obtained by the electrolysis of lead acetate baths containing ammonium perchlorate and peptone[1] were much better than had been obtained previously from any lead salt of a weak acid. The cathodes were smooth during the first part of the deposition, but as the thickness of the deposit increased the cathodes became more and more rough. All of these deposits were brittle. Cathodes having a thickness of 0.5 cm. on a side were so rough that they were not saved as samples. These baths lacked something, and the deposits were not satisfactory. This paper gives the results with some new addition agents which produce the best lead deposits that have ever been obtained from the lead salt of any weak acid.

Manipulation.

The experiments were made in essentially the same manner as has been described previously in another paper.[1] Beakers of 250 cc. capacity were used as electrolyzing vessels. The volume of the bath was 200 cc. in each case. A cathode of 25 sq. cm. area on a side 4.2 by 6 cm. or 5 by 5 cm. (1.6 by 2.3 in. or 1.9 by 1.9 in.) made of thinly rolled lead was suspended from a glass rod by two narrow strips of lead. Two anodes 3.5 by 4 by 0.5 to 1 cm. (1.3 by 1.5 by 0.19 to 0.4 in.) were suspended in each beaker by small copper wires passing through a hole in

[1] Mathers, Trans. Amer. Electrochem. Soc., 24, 315 (1913).

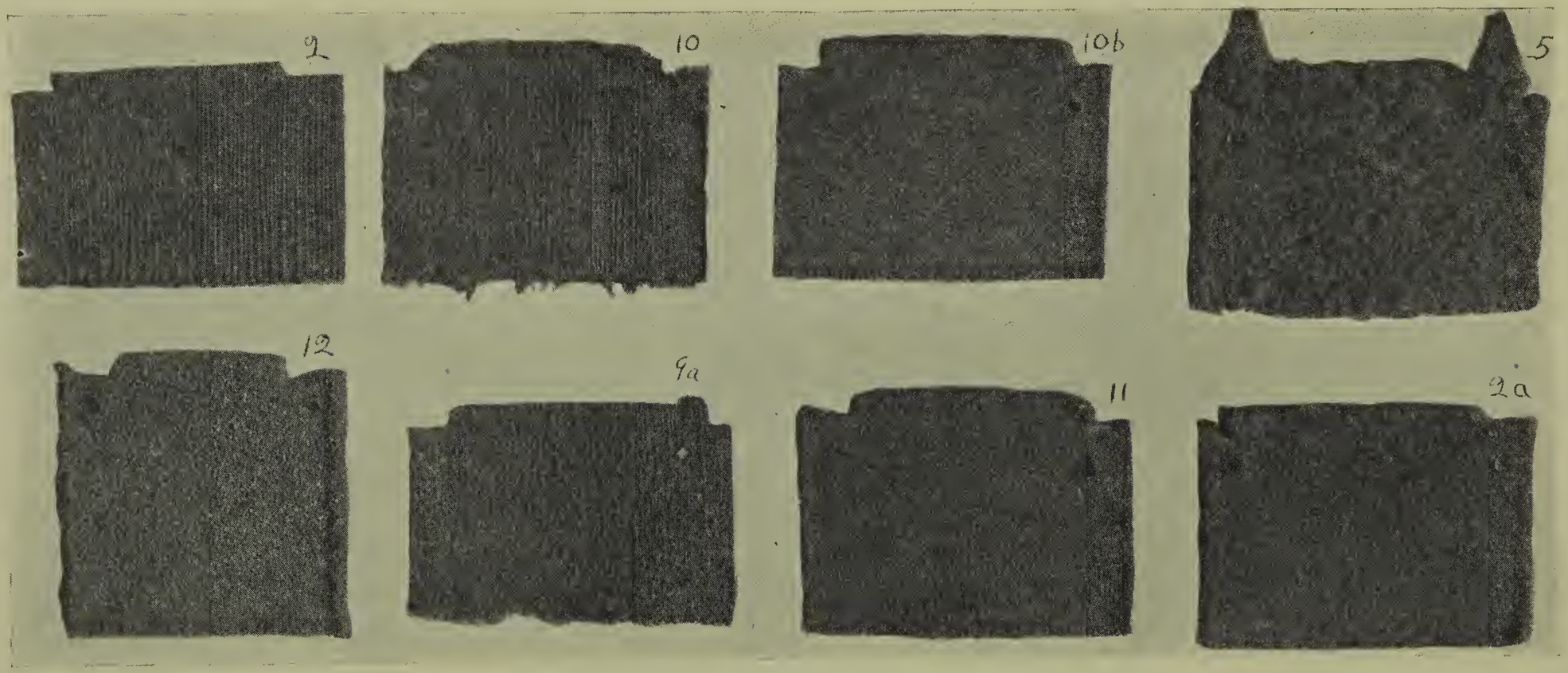

FIG. 1.—*Addition Agents from Poplar Wood.*

Each 200 cc. bath contained 20 g. lead acetate, 8 g. ammonium perchlorate, unless otherwise stated, and other substances as given below. Complete descriptions of these baths can be found under the corresponding numbers in the text of this article.

2. The addition agent was prepared by boiling 10 g. of the wood in 20 cc. of 50 percent acetic acid. The striations were caused by too much addition agent.

10. Five grams of the wood were boiled in 50 cc. of 40 percent acetic acid for 19 hours. No ammonium perchlorate was added, hence the loose trees and the striations.

10*b*. Same bath as in 10, except that 10 g. of ammonium acetate was added. The deposit is not as good as with ammonium perchlorate.

5. Same as 2a, except that sap wood was used in place of the yellow heart wood.

12. Five grams of the wood was boiled for 10 hours in 100 cc. water. Five cc. of acetic acid was added to the bath.

9*a*. Five grams of the wood was boiled for two hours in 100 cc. of 5 percent acetic acid.

11. Addition agent prepared as in 10. No acetic acid was present.

2*a*. A normal cathode such as should be obtained by using an addition agent prepared by boiling poplar wood in strong acetic acid. Both ammonium perchlorate and acetic acid are needed for the best deposits.

the center of each anode. Each anode was wrapped in filter paper to prevent anode slimes from reaching the cathode. Unless otherwise stated, each bath contained 10 percent lead acetate and 4 percent ammonium perchlorate with varying amounts of acetic acid and various addition agents. The current density was 0.4 amp. per sq. dm. (3.6 amp. per sq. ft.).

Acetic Acid Extracts of Vegetable Materials as Addition Agents.[2]

In the former research, an addition substance was prepared by boiling casein in acetic acid. The deposits obtained by addition substances prepared in this way were promising, but none was satisfactory. It was thought that other organic materials when treated in the same way with acetic acid might give satisfactory addition agents. There is no mention in the literature, so far as the author has been able to investigate, concerning the use of addition substances prepared by boiling organic material in acetic acid. In one book, the name and author of which has been forgotten, there is a statement that new wooden electroplating tanks produce an action upon the metal deposits. This indicates that wood contains some active addition substance which dissolves in the electrolyte. Tannin, licorice and other water extracts of vegetable substances have been used in many instances.

Poplar Wood (Liriodendron Tulipefera, L.).—Seasoned poplar wood, either as shavings or small splints or sawdust, was boiled with acetic acid. The solutions or extracts thus obtained were of a yellow color, being darker the longer the extraction or boiling was continued and the more concentrated the acetic acid. This extract produced a yellow or brown precipitate when it was added to the lead acetate bath. This precipitate was filtered out in most cases, the filtrate being the finished bath ready for electrolysis. These baths had a nice yellow color and the odor of poplar wood. Under the best conditions for preparing the addition substance, the deposits were excellent. Samples of cathodes obtained by the use of extracts prepared in various ways are shown in Fig. 1. The specific gravity of the lead in one of the best cathodes was 11.27, as compared with 11.37 for pure lead.

The heating with the acetic acid was done in an apparatus

[2] Mr. G. W. Gates assisted in this part of the work.

consisting of a flask and a reflux condenser which fitted into the neck of the flask, so that no rubber, cork or grease was used.

1. Ten grams of the yellow heart wood of poplar in small split pieces was boiled for 17 days in 60 cc. of 33 percent acetic acid. The pieces of wood did not dissolve, but they became dark in color and soft. It is probable that a large amount of the water had distilled out of the apparatus and that very concentrated acid was present during the latter part of the extraction. When water was added to rinse out the flask, a precipitate was formed. The deposit of lead was very good, being solid, smooth and of a fine metallic color.

2. Ten grams of the yellow heart wood of poplar was boiled for 42 hours with 40 cc. of 50 percent acetic acid. The deposit was hard, firm and very free from crystals. The cathode showed peculiar vertical ridges which ended in drop-like masses of metal at the bottom of the cathode. This indicated that too much addition substance was present. The bath was diluted with lead acetate and ammonium perchlorate solution until the volume was 400 cc. The deposits from this bath were very smooth and did not show any of the striations or vertical ridges.

3. The residue of wood from experiment 2 was boiled for 102 hours with 40 cc. of 50 percent acetic acid. The deposit was a little rough in places and not as good as in experiment 2.

4. The residue from experiment 3 was again boiled with 40 cc. of 50 percent acetic acid. The deposit was not good, being rough and brittle, but no trees or long, loose crystals formed.

5. The addition substance was prepared as in experiment 2, except that the sap wood of the poplar was used instead of the yellow heart wood. The time of boiling was for 114 hours. The deposit was solid and finely crystalline, but slightly rough. The sap wood seemed not to give as good a deposit as the heart wood.

6. Five grams of the heart wood of poplar was placed in a filter paper extraction thimble which was suspended by a glass rod in an apparatus similar to a Wiley extractor. Fifty grams of glacial acetic acid was used, and the extraction was continued for 46 hours. There seemed to be some decomposition during the boiling, since there was a separation of solid material upon the flask during the extraction. The acetic acid extract was transferred to a flask and distilled to near dryness under reduced

pressure. Water was added and the material was added to the bath. The deposit was finely crystalline but rough.

7. A second extraction of the residue from experiment 6 for 70 hours gave a still rougher deposit than in experiment 6.

8. Poplar wood was extracted as in experiment 6, but for only one hour. The deposit was firm and a little rough, but better than in experiment 6.

9. The addition agent obtained by treating 5 g. of the heart wood of poplar with dilute acetic acid gave in every case a deposit having a peculiar rough surface. This characteristic deposit seemed to be independent of the time or temperature of the extraction, as can be seen from the following experiments which show the variations of conditions.

a. Five-gram portions of the poplar wood were boiled for 2 hours, for 15 minutes and for 2 minutes, respectively, in 100 cc. of 5 percent acetic acid.

b. The wood and the 100 cc. of 5 percent acetic acid remained together at room temperature for 46 hours.

c. The wood was moistened with 5 cc. of glacial acetic acid, and warmed for one minute in a water bath. Ninety-five cc. of water was added. The mixture was stirred, filtered, and the extract was used.

d. The wood was treated as in experiment *c,* except that the wood was moistened with 10 cc. of 50 percent acetic acid and 90 cc. of water was added.

e. The wood was treated as in experiment *d,* except that the mixture, after being moistened with the 10 cc. of 50 percent acetic acid was allowed to stand 46 hours before the water was added.

In all of these cases the deposits had the peculiar rough surfaces. If a new cathode was placed in the bath after electrolysis for a day, a better but still a rough deposit was obtained. The general appearance of these second deposits or cathodes seemed to show a deficiency of addition agent.

10. Five grams of poplar wood was boiled in 50 cc. of 40 percent acetic acid for 19 hours. This extract was added to a bath containing the 10 percent lead acetate but no ammonium perchlorate or other salt. The deposit was striated, with loose trees or projections on both the top and bottom. The addition of 5 percent ammonium acetate caused the deposit to become firm

but crystalline, consequently a thick cathode would have been very rough.

11. Wood extract prepared exactly as in experiment 10 was evaporated to dryness under reduced pressure in order to remove all the acetic acid. This residue was taken up in water as completely as possible and added to a bath containing the lead acetate and ammonium perchlorate but no acetic acid. The deposit was solid and firm but crystalline, consequently a thick cathode would have become rough.

12. Five grams of sawdust of poplar wood was boiled in 100 cc. of water for 10 hours. This extract was added to a bath containing lead acetate, ammonium perchlorate and 2.5 percent acetic acid. The deposit was crystalline, but was very smooth and firm.

Pine Wood.[3]—13. Five grams of pine wood in small splints was boiled in 50 cc. of water for 74 hours. The final bath had a pale yellow color. The lead deposit was dark and spongy. A similar experiment in which 2.5 percent of acetic acid was added to the bath gave a deposit which was rough, being covered with projecting crystals. The deposit seemed to be very good for the first few hours.

14. Five grams of pine wood was boiled with 25 cc. of glacial acetic acid for 74 hours. A brick-red precipitate was formed when this extract was added to the bath. The cathode was smooth in the center, but fine, thread- or hair-like growths formed on the edges. This cathode was very different from the deposit obtained with poplar wood under similar conditions.

15. The residue of pine wood from experiment 14 was boiled in 40 cc. of 50 percent acetic acid for 312 hours. The deposit was very rough and brittle.

Linden Wood (Tilia americana).—16. Five grams of the sawdust of linden wood was extracted in a form of Wiley extractor with 50 cc. of glacial acetic acid for 46 hours. The extract thus obtained was evaporated to dryness under reduced pressure. Five cc. of acetic acid was added to dissolve the residue of extract in the flask. The solution thus obtained was introduced into the lead bath. The deposit was firm but rough and not as good as when the extract from poplar wood was used.

[3] The species of pine is not known.

Black Oak Wood (Quercus nigra).—17. Five grams of the sawdust from black oak was extracted for 3.5 hours with 50 cc. of glacial acetic acid in a form of Wiley extractor. The extract was distilled to dryness under reduced pressure, and then taken up in 5 cc. of glacial acetic acid. The deposit was as good as when the extract from poplar was used.

18. The residue of sawdust remaining from experiment 17 was extracted again as in that experiment. The deposit was rougher and not as good as was obtained by the first extraction.

Black Walnut Wood (Juglans nigra).—19. Addition agents prepared by extracting black walnut wood produced as good deposits as those from poplar.

Tannin.—20. Five grams of tannin was boiled in 10 cc. of glacial acetic acid for 168 hours. A very heavy precipitate was produced when this mixture was added to the lead bath. Twenty grams more of lead acetate was added to replace that lost in the precipitate. The deposit was spongy. This shows that the active substance extracted from wood is not tannin.

Oil of Sassafras.—21. Five grams of oil of sassafras was boiled with 25 cc. of glacial acetic acid for 46 hours. The solution was then evaporated nearly to dryness under reduced pressure, and 5 cc. of acetic acid was added to take up the residue. The deposit consisted of very loose crystalline leaves, and, on the whole, was not much different from deposits where no addition substances had been used.

Essential oils had been found to be of little use in lead acetate baths, but it was thought that the boiling with acetic acid might in some way effect a change in the oil. This experiment is taken to show that the beneficial action of the acetic acid extracts from woods is not due to essential oils.

Leaves.—22. Ten grams of green leaves from a beech *(Fagus americana)* tree was boiled for several hours in a bath containing 2.5 percent acetic acid. After filtering, the bath was electrolyzed. The deposit was dark and spongy.

Licorice.—23. One gram of dried licorice extract was dissolved in water and added to a bath containing 2.5 percent acetic acid. A heavy precipitate was formed, which was filtered out. The deposit was rough, and finally became spongy in spots.

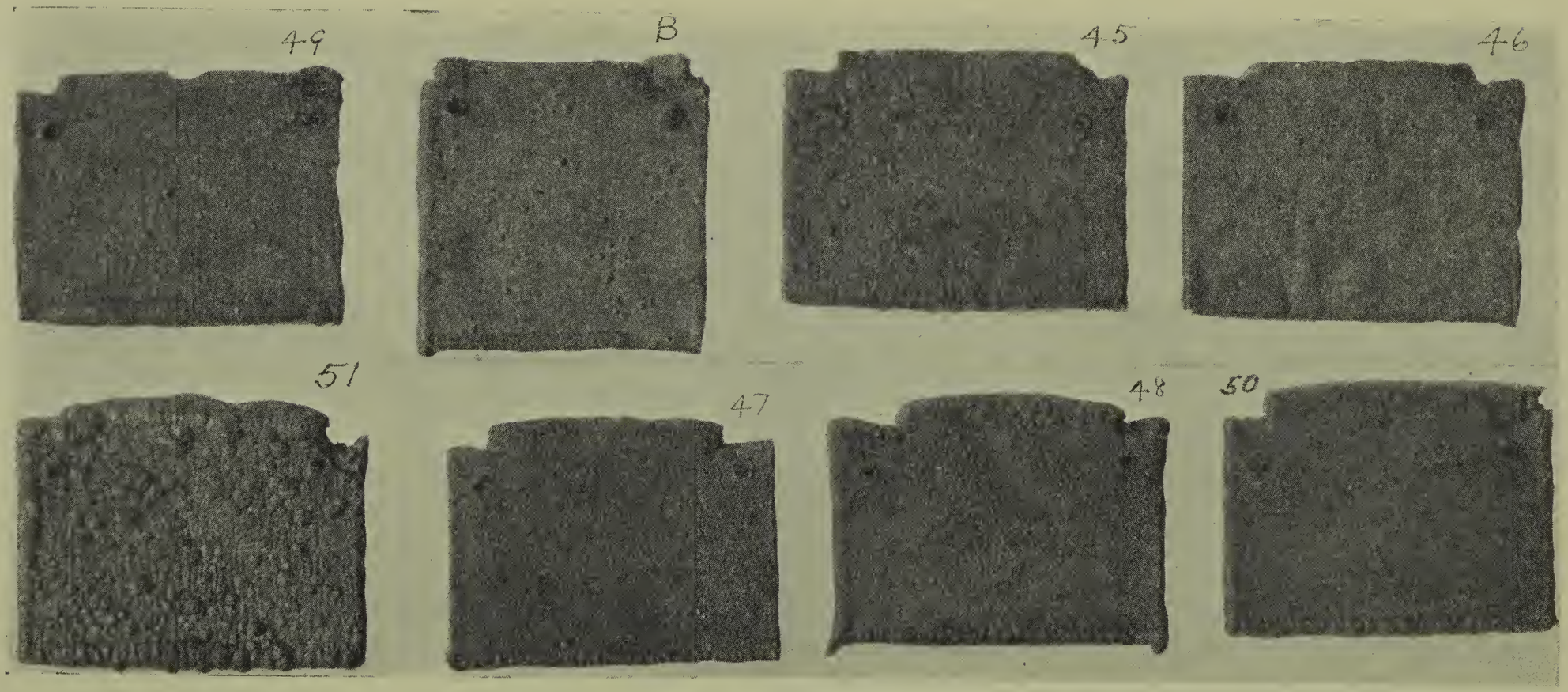

FIG. 2.—*Addition Agents Prepared from Aloes.*

Each 200 cc. bath contained 20 gm. lead acetate, 8 gm. ammonium perchlorate, 5 to 10 cc. acetic acid and an addition agent as described below:

49. The insoluble gum or residue from extraction of 2.5 gm. of Barbadoes aloes in warm water.
B. One gram of the insoluble residue obtained in the commercial extraction of Curaçao aloes in the manufacture of aloin.
45. One gram Socotrine aloes was added to a bath which was warmed for several hours before electrolysis.
46. Like 45, except that Barbadoes aloes was used.
51. Two and five-tenths grams of Barbadoes aloes was extracted in a Soxlet extractor with alcohol. The addition of water to this extract and evaporation to remove the alcohol precipitated a gum which was used in this experiment.
47. One gram of purified aloin, the active principle in aloes.
48. One gram of Barbadoes aloes was extracted with water in a Soxlet extractor.
50. One gram of Barbadoes aloes was extracted in a Soxlet extractor with alcohol. This extract was used after the alcohol had been removed.

24. Licorice root or the dried extract boiled with 50 percent of glacial acetic acid gave addition substances which produced coarsely crystalline deposits.

Pyrogallol.—25. One percent of pyrogallol in a bath containing 5 percent acetic acid gave a cathode which was smooth in places but covered with light, feathery crystals in other places. There was a heavy precipitate in the bottom of the beaker, due to a reaction between the lead acetate and the pyrogallol. The deposit finally became spongy.

Extracts from Aloes as Addition Agents.

Two and five-tenths grams each of Socotrine aloes, Barbadoes aloes, gum Guaiac and gum Catechu were separately boiled in 20 cc. of glacial acetic acid for 16 hours. The extracts were evaporated to dryness under reduced pressure, and then the residues of the extracts were redissolved in 5 cc. of acetic acid, respectively. These extracts were used in the baths. The deposits in the cases of the Catechu and Guaiac were rough, with loose projecting crystals. The deposits obtained with the aloes were excellent. Experiments with extracts from wood and other organic materials were discontinued because the aloes was a very cheap material from which the addition agent could be prepared very easily.

Aloes has been tried as addition agent in perchlorate baths.[4] The photographs of these cathodes show that firm, smooth rods or clubs of metal were formed upon the cathodes. Except for these projections, the deposits were very smooth and finely crystalline. These peculiar deposits may have been caused by a precipitate in the bath. The deposits from acetate baths, with aloes as addition agent, never showed any of these peculiarities, although precipitates were often present.

Aloes is the inspissated or thickened juice which has exuded or been pressed from the leaf of various species of aloes. Barbadoes, Curacao and Socotrine are the names given to the aloes obtained from different regions. The plants from which these aloes are obtained belong to different species, but the chemical substances in the different aloes are very closely related. The

[4] Mathers and Overman, Trans. Amer. Electrochem. Soc., **21**, 327 (1912).

active principle is aloin, which is used medicinally as a purgative. The prices of aloes as given in drug lists are from ten to twenty cents per pound.

26. Five baths, each containing 2.5 percent of acetic acid, were treated with 0.4, 0.3, 0.2, 0.1 and 0.05 g., respectively, of Socotrine aloes. The baths were then warmed for an hour on the hot plate, and any undissolved aloes was filtered out. The deposit from the bath containing 0.4 g. aloes was the best, although it was rough and nodular. The cathodes were poorer the smaller the amount of aloes, until with 0.05 g. loose, flaky crystals formed upon the edges. The use of 5 percent of acetic acid in a similar series of baths greatly improved the deposits.

41. A few of the above experiments were duplicated, except that the mixtures were heated for several hours instead of one hour. The deposits were smoother and better than in the above experiments.

44. One gram of Socotrine aloes and 2.5 percent of acetic acid were added to a bath, which was then heated for several hours. The deposit was excellent, being finely crystalline and free from loose projections. The cathode was slightly nodular. The increase of the acetic acid to 5 percent in a similar bath did not seem to improve the deposit in any way.

46. The use of Barbadoes aloes in place of Socotrine aloes in a series of experiments similar to those described in 26 produced sightly better deposits. This improvement was more marked in the experiment using 0.5 g. of the aloes.

After these baths had been electrolyzed for a long time the deposits became rough and, finally, spongy. Baths, after continuous electrolysis for a month, were neutral to litmus. The deposits did not become good even after the addition of 15 cc. of acetic acid. One gram of Barbadoes (or Socotrine) aloes was added to each bath, and the baths warmed for several hours and filtered. The deposits were as good as when the baths were new.

47. Four-tenths gram of pure aloin, the active material in aloes, was used in a bath containing 2.5 percent acetic acid. The aloin dissolved completely upon warming the bath, and filtration was not necessary as in the cases where the crude aloes was used.

The deposit was not as smooth and free from roughness as when the crude aloes was used.

48. Ten grams of Barbadoes aloes was extracted with water in a Soxlet extractor. One-tenth of the extract thus obtained, equivalent to one gram of the aloes, gave a deposit which was about equal to that obtained when the pure aloin was used. The bath contained 2.5 percent acetic acid.

49. A brown tarry gum separated upon the sides of the Soxlet flask during the extraction in experiment 48. This gum was dissolved in 5 cc. acetic acid and added to a bath. The bath thus prepared was electrolyzed without removal of the precipitate. The deposit was smooth, very free from nodules, and the best that had been obtained yet.

50. Ten grams of Barbadoes aloes was extracted with alcohol in a Soxlet extractor. The extract was a beautiful wine-red color, but it soon darkened in the flask. This extract was treated with water and evaporated until the alcohol was removed. A quantity of this water solution equal to one gram of the aloes was added to a bath containing 2.5 percent acetic acid. The deposit was about like that obtained by extraction with water as described in experiment 48.

51. The gum or tarry matter that precipitated from the alcohol extract when water was added and the mixture boiled, in experiment 50, was dissolved in 5 cc. acetic acid and added to a bath. The deposit was excellent, being smooth, finely crystalline and free from all loose projections.

52. Ten grams of Barbadoes aloes was shaken with warm water in a 250 cc. graduated flask until the soluble material had dissolved. The volume was then made up to the mark. This solution was allowed to stand over night so that the gum or tarry matter would settle. The clear solution thus obtained produced no precipitate when it was added to a lead bath containing 2.5 percent of acetic acid. The addition substance prepared in this simple manner gave as good results as when made by more elaborate methods.

53. The Barbadoes aloes was boiled with water for 21 hours. This boiling did not seem to change the addition substance in any way, since the cathode was not different from the one obtained in experiment 52.

54. Socotrine aloes was boiled with water for 19 hours. The cathode deposit did not differ materially from the ones obtained by the use of the Barbadoes aloes in 52 and 53.

55. The insoluble tarry matter that adhered to the flasks in experiments 52, 53 and 54 was dissolved in acetic acid and used as addition agent. With the tarry matter obtained from 2.5 grams of the Barbadoes or Socotrine aloes and 2.5 percent acetic acid the baths gave excellent deposits. The baths were not filtered before electrolysis.

56. If baths like those described in 55 were filtered before electrolysis, the deposits became rough after a short time. This was taken to mean that the precipitate of the aloes residue in the bath gradually dissolved during the course of the electrolysis and that this precipitate contained the important addition substance.

The following table shows the effect of varying the concentration of the bath. A current density of 0.8 amp. per sq. dm. (7.2 amp. per sq. ft.) was used. The addition substance was prepared by dissolving a quantity of the aloes in warm water. The clear liquid, after the tarry matter had settled, was used.

	Volts	Percent Lead Acetate	Percent Ammonium Perchlorate	Percent Acetic Acid	Percent Aloes	Description of Deposit
57	0.39	10	4	2.5	1.5	Much rougher than with lower current density.
58	0.28	10	8	2.5	1.5	Smoother than with less ammonium perchlorate.
59	0.44	20	4	2.5	1.5	No better than when less lead was used.
60	1.8	10	0	2.5	2.0	Loose, projecting crystals on the edges.
61	. .	10	0	0	0.5	Dark, spongy and non-adherent.

The results given in this table show that the presence of ammonium perchlorate is very important and that the use of 8 percent instead of 4 greatly improves the deposit. Any increase in the amount of lead acetate above 10 percent is of no importance. With neither free acid nor ammonium perchlorate the deposit was very spongy. With lower current densities these variations in concentration are of less apparent effect.

Substitutes for Ammonium Perchlorate.—Ammonium perchlorate is a more expensive salt than numbers of others. The necessary restriction of salts for use in these baths to those that form soluble lead compounds leaves only a comparatively few salts. These experiments were tried upon baths containing 10 percent lead acetate, 2.5 percent acetic acid and 0.5 percent of Barbadoes aloes, as a clear water extract.

62. Whenever the bath contained only lead acetate, addition substance and acetic acid, the cathodes had loose projecting crystals or trees upon the edges and on the sides of the cathodes where the current density was a little high. At other places the deposits were smooth and of apparently good quality.

63. The use of 2 percent potassium perchlorate gave a fair deposit, but one that showed too many rough projecting crystals. A thick, smooth deposit could not have been obtained. The difficulty was perhaps due to the low concentration of potassium perchlorate, which is too insoluble to form a 4 percent solution.

64. Four percent sodium perchlorate did not give as good a deposit as the ammonium perchlorate, but there was very little difference between the two. The conclusion must be drawn that the ammonium ion assists slightly in some way in improving the deposit.

65. Four percent of ammonium acetate gave a fair deposit. There was a great tendency for the cathode to become rough. This bath can be used with good results for ordinary plating operations where only very thin deposits are desired. Samples of thin plates of lead from this bath have a nice, bright crystalline appearance. At 0.8 amp. per sq. dm. this bath did not give a good cathode even when the deposit was very thin.

66 and 67. Four percent potassium chlorate and 4 percent sodium nitrate separately gave baths from which excellent deposits were obtained. These deposits were equal in every way to any that were obtained during this work. These cathodes disprove the older idea that solid, smooth deposits of lead cannot be obtained from oxidizing solutions. Unfortunately these baths are so unstable that after a time good deposits can no longer be obtained. The subject of plating from lead nitrate baths will be fully treated in another paper in these Transactions.

68. Four percent ammonium fluoborate gave a fair deposit

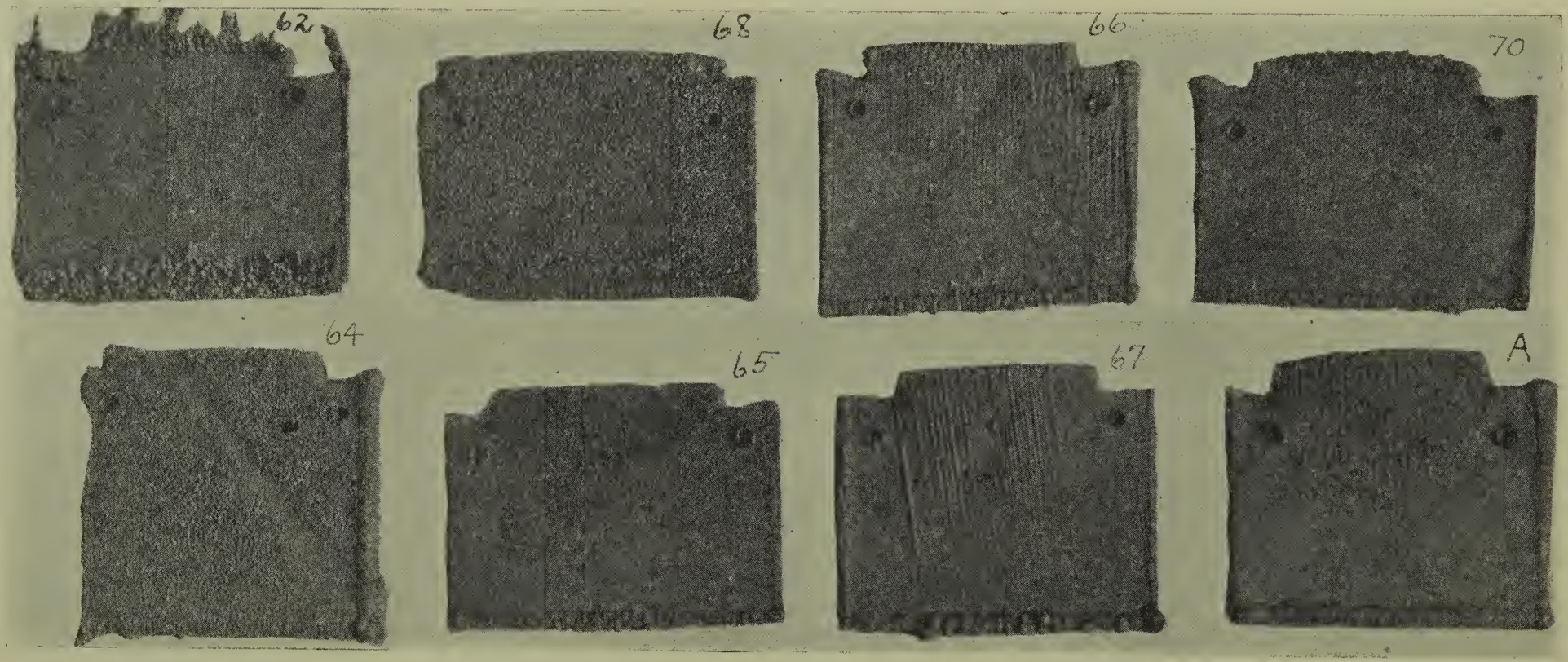

FIG. 3.—*Substitutes for Ammonium Perchlorate.*

Each 200 cc. bath contained 20 g. lead acetate, 5 cc. acetic acid, the aqueous extract from 1 g. Barbadoes aloes and a quantity of some salt as given below:

62. No salt was present in this bath, consequently projecting trees were formed.

68. Eight grams ammonium fluoborate.

66. Eight grams potassium chlorate. An excellent deposit was produced in the presence of this oxidizing agent.

70. Eight grams ammonium ethyl-sulphate.

64. Eight grams sodium perchlorate. The deposit is not as good as with ammonium perchlorate.

65. Ten grams ammonium acetate.

67. Eight grams sodium nitrate. Again, a strong oxidizing agent assisted in the formation of an excellent deposit.

A. Eight grams ammonium perchlorate. This cathode is placed in this figure for comparison.

which was very crystalline. The bath became poor in lead because the anode did not dissolve well in the bath, and consequently the upper portion of the cathode was spongy. This insolubility of the anode was caused perhaps by the decomposition of the ammonium fluoborate, whereby lead fluoride formed upon the anode as an insoluble non-conducting cover.

69. Sodium fluosilicate is too insoluble to make a 4 percent solution. The bath was saturated with the sodium fluosilicate, and 5 percent of acetic acid was used in order to prevent as much as possible the decomposition of the fluosilicic acid radicle. Nevertheless, the anode became covered with a grey adherent slime, and a bulky precipitate formed in the bath. A deficiency in lead was shown by the black spongy deposit on the top of the cathode. Ten percent more lead acetate was added. The deposit was no longer black or spongy, but became crystalline and rough. It seemed impossible to make use of these complex fluorides on account of their instability.

70. Four percent ammonium ethylsulphate gave a finely crystalline deposit which was a little rough but firm.

An examination of these cathodes shows that the best addition substance is the tarry wax or gum that remains insoluble when aloes is extracted with water. The water-soluble matter from aloes and the pure aloin itself have excellent beneficial action when used as addition substances, but this insoluble residue surpasses all of them. Some samples of the insoluble residues remaining after the extraction of Curacao aloes in the commercial manufacture of aloin have been obtained through the kindness of the New York Quinine and Chemical Company and of Sharp and Dohme of Baltimore. A few experiments have shown that these residues, which are available in large amounts, are as beneficial addition agents as the residues obtained from Barbadoes aloes on the small scale laboratory experiments. More extended experiments are being made with these residues, consequently they need not be discussed more in this paper.

Discussion and Summary.

This paper describes experiments with some new addition agents by means of which smooth, finely crystalline deposits of lead may be obtained from acetate baths. These deposits are

much superior to those obtained with peptone as an addition agent.

For each liter (1.056 quarts) of bath there should be used 100 g. lead acetate, 40 g. ammonium perchlorate, 25 to 50 cc. of glacial acetic acid and addition substance prepared by one of the following methods:

1. Boil for two hours 25 g. of poplar wood sawdust in the acetic acid diluted with an equal volume of water. Add this extract to the solution of the lead acetate and the ammonium perchlorate. Filter, dilute to one liter and electrolyze.

2. Boil 50 g. of the sawdust of poplar wood in 500 cc. of water for 8 to 10 hours. Filter and add this aqueous extract to the bath. Dilute to one liter and electrolyze without filtering.

3. Treat 5 g. of aloes with 200 cc. of warm water and shake until the soluble material has dissolved. Let stand until the insoluble tar or gum settles and adheres to the sides of the vessel. Add this clear aqueous extract to the lead bath and electrolyze without filtering.

4. Dissolve the insoluble tar or gum (mentioned in the preceding paragraph) obtained from the extraction of 12.5 g. of aloes with water, in the glacial acetic acid. Add this solution to the bath and electrolyze without filtering. No better addition agent than this was found.

5. Dissolve 10 g. of the residue that remains after the commercial extraction of aloes in the manufacture of aloin, in the acetic acid and add to the bath. Electrolyze without filtering.

The addition substance extracted from poplar wood by boiling in strong acetic acid gives very good results. The loss of acetic acid in the residue of wood is a serious disadvantage if used commercially. The deposits are very good, but they have no advantage over those obtained by the use of aloes. The addition agent produced by boiling the wood in dilute acetic acid (5 percent) produced rough, unsatisfactory deposits. The residue of wood remaining from one extraction when extracted again does not give as good deposits as the first time. Sap wood of the poplar seemed to be less valuable than the heart wood. The addition substance extracted from the wood is not tannin or essential oils, since these substances are of little effect in these lead baths. Some woods, as pine, are not suited for the prepara-

tion of addition agents. There are some other woods just as good as the poplar.

Merely boiling the wood in water gives an addition substance which produces a very crystalline, firm, but uneven deposit. This addition substance could be used in plating work where only a very thin deposit is required. It seems to the author that there are possibilities in these aqueous extracts from woods. This part of the work was covered very briefly.

The use of the aqueous extract of aloes is not recommended. although very good deposits may be obtained with it, because the use of aloes residue gives so much better results. Very many methods of preparing the addition agent from the aloes were tried, but the simple method given above was as good as any. The two aloes, Barbadoes and Socotrine, seemed to be about equally beneficial.

The residue from aloes which is insoluble in water is the best addition substance that was found. Thicker, smoother deposits could be obtained with it than with any other addition agent. This residue is soluble in hot strong acetic acid, but a large part of it reprecipitates when this solution is added to the bath. If this precipitate is filtered out before electrolysis, the deposit becomes rough much sooner than if it is allowed to remain in the solution. It is thought that this precipitate gradually redissolves in the bath during the electrolysis. More extended experiments are to be made upon the use of this aloes residue as addition agent in metal deposition.

Good deposits could be obtained for a short time from a new bath which did not contain free acetic acid. In the absence of this free acid a precipitate gradually formed in the solution which perhaps caused the deposit to become rough. The deposits became spongy when the baths became neutral in reaction after continued electrolysis.

The presence of ammonium perchlorate or similar salt was necessary for the formation of a good smooth deposit. Good thin deposits could be obtained when ammonium acetate was used, but thick deposits became rough. Ammonium perchlorate was the most satisfactory salt that was tried. Sodium nitrate or potassium chlorate gave excellent deposits, but they seem to be too unstable for practical purposes. Better deposits could be

obtained by increasing the ammonium perchlorate to 8 percent, but this greatly increases the cost of the bath.

A current density of 0.4 amp. per sq. dm. (3.6 amp. per sq. ft.) must be used for thick deposits. For thin deposits in plating 0.8 amp. per sq. dm. may be used. With the higher current density, ammonium acetate cannot be used in place of the ammonium perchlorate.

The current efficiencies at the anode and at the cathode are near 100 percent. The excellent corrosion of the anodes is proved by the fact that the copper wires attached to the anodes in the solution were not dissolved. The specific gravity of the deposited lead was 11.27.

More of the addition substance and more acetic acid must be added to the bath during the course of the electrolysis.

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A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 2, 1914, President F. A. Lidbury in the Chair.

ELECTRODEPOSITION OF LEAD FROM LEAD LACTATE AND LEAD FORMATE SOLUTIONS

By Frank C. Mathers and Barrett W. Cockrum.

Lead has been deposited from lead lactate[1] and formate[2] solutions, but the deposits were spongy, non-adherent and of no value either for quantitative work or plating purposes. The formation of these spongy, non-adherent deposits has given credence to the false idea that solid electrodeposited lead can not be had from the solutions of the lead salts of weak acids.

These experiments were made to determine whether or not lead could be deposited in a solid condition by the electrolysis of lead lactate and lead formate solutions according to the method previously used for the electrolysis of lead acetate solutions.[3]

Apparatus and Solutions.—Lead lactate was prepared by neutralizing lactic acid with litharge (PbO). Lead formate was prepared by neutralizing formic acid with lead carbonate. The residue of lead formate was purified by crystallization from hot water.

Two lead anodes 4 x 4 x 0.5 cm. (1.56 x 1.56 x 0.2 in.) were suspended by copper wires in opposite sides of a 225 cc. beaker. These anodes were wrapped in filter paper in order to keep the slime out of the bath. A cathode 6 x 4.2 cm. (2.3 x 1.6 in.), made of thinly rolled lead, was hung between the anodes. The volume of each bath was 200 cc.. The current was 0.4 amp. per sq. dm. (3.6 amp. per sq. ft.).

Each lactate bath contained 4 percent of lead as lactate and 4 percent of ammonium perchlorate, and each formate bath contained 2 percent of lead formate and 4 percent of ammonium perchlorate, unless otherwise stated. The other things which were added to the baths are stated in each case. The experiments were all run at room temperature.

[1] Jordon, Z. Electrochemie, **2**, 140 (1905).
[2] Warwick, Chem. News, **66**, 275 (1892).
[3] Mathers, Trans. Amer. Electrochem. Soc., **24**, 315 (1913).

LEAD LACTATE BATHS

Effects of Addition Substances.

No Addition Substance.—The deposit from the bath containing only lead lactate was very crystalline with long needles which soon short-circuited the bath. The addition of 4.5 percent of lactic acid gave essentially the same crystalline deposit, which soon short-circuited the cell. These deposits are characteristic of lead as usually obtained from solutions of lead acetate or other lead salts of weak acids. The addition of 4 percent of ammonium perchlorate to each of the above baths gave deposits which were slightly less crystalline, thus showing the beneficial effect of ammonium perchlorate. The presence of free lactic acid seemed to be without any beneficial action.

Glue.—The addition of one percent of glue, either with or without 4.5 percent of free lactic acid, gave deposits which were less crystalline than without the glue. After electrolysis for 48 hours the cathodes were a mass of crystals. This shows that the glue had some effect, since without it the electrolysis of a bath for this length of time was impossible because of the short-circuiting of the cell. After the addition of 4 percent of ammonium perchlorate to each of these baths, the deposits became finely crystalline, but changed in a short time, becoming black and spongy and falling from the cathode to the bottom of the beaker. The formation of such a loose, spongy deposit quite often occurs when too much addition substance has been added. Another experiment using 0.2 g. glue with the ammonium perchlorate, but without free lactic acid, gave a fairly smooth deposit, which, however, became rough before it attained any considerable thickness. The deposit was covered thickly with projecting pointed crystals. These experiments show the great improvement produced by the addition of the ammonium perchlorate along with the glue, and also that the glue does not have the properties which supplement those of the other acid ions in the baths in a way to produce smooth deposits.

Clove Oil.—Four drops of clove oil[4] was without effect either in the presence of free acid (4.5 percent) or ammonium per-

[4] An Excellent Addition Agent in Lead Perchlorate or Lead Fluosilicate Baths. Mathers and Overman, Amer. Electrochem. Soc., **21**, 33 (1912).

chlorate or both. The deposits were as crystalline as when no addition agent was used.

Nicotine.—Four drops of nicotine was of no value, the deposit being apparently as crystalline as when no addition substance was used.

Peptone.—A bath containing 0.5 percent of peptone and 4 percent of lead as lactate gave a black spongy deposit which fell from the cathode and settled to the bottom of the beaker. The addition of 4 percent of ammonium perchlorate to this bath caused it to give a firm, solid but rather brittle deposit. Deposits from solutions containing less than 0.5 percent of peptone had rough crystalline projecting edges. The deposit from the solution containing 0.125 percent of peptone was free from the feathery crystals on the face of the cathode which is a distinction from the deposits gotten from the solutions containing no peptone, in which cases feathery crystals covered the faces of the cathodes. The deposits were, however, rough on the edges. The deposit from the bath containing one percent of peptone was not appreciably better than those with 0.5 percent, hence the latter amount was considered to be the proper quantity.

Deposits were obtained from baths containing 4, 3, 2 and 1 percent of ammonium perchlorate, respectively, and 4 percent of lead as lactate and 0.5 percent of peptone in each case, but without free lactic acid. The deposit from the bath containing 4 percent of ammonium perchlorate was much superior to the others. With one percent of ammonium perchlorate the deposit was very dark and rough, especially on the edges. It was also very brittle. These deposits are shown in Fig. 1. Duplicate experiments with baths containing 5 percent of lactic acid gave no better results. Deposits from baths containing from 8 to 2 percent of lead as lactate, with 4 percent of ammonium perchlorate, 0.5 percent of peptone and one to two percent of free lactic acid were all about equal in quality. The decrease in the concentration of lead was almost without effect upon the deposits; however, the bath with 4 percent of lead was judged to be the best to use, because those with higher lead content were not enough better to make important the higher concentration. These cathodes are

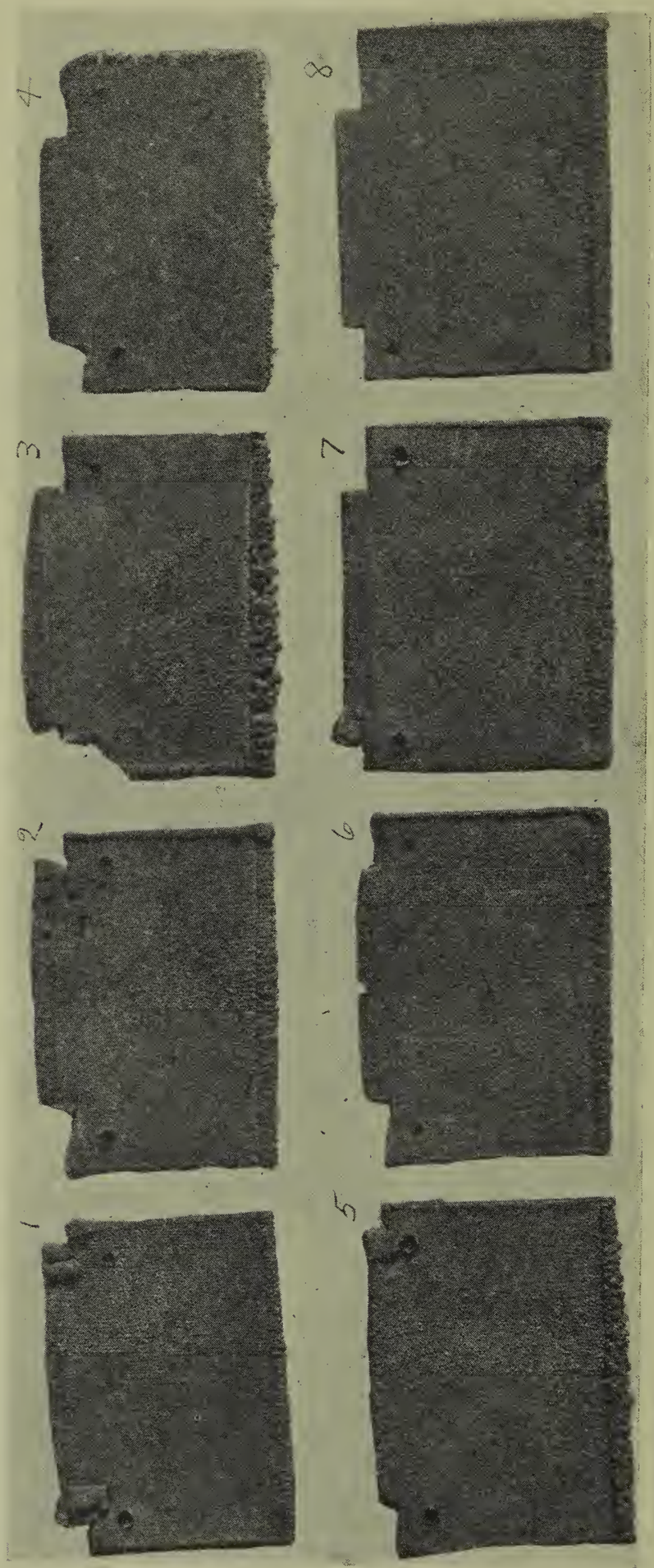

FIG. 1.—*Deposits from Lead Lactate Baths.*

Cathodes 1, 2, 3 and 4, were obtained from baths containing 4 percent of lead as lactate, 0.5 percent peptone, but no free lactic acid and 4, 3, 2 and 1 percent ammonium perchlorate respectively. This series shows the importance of ammonium perchlorate. Cathodes 5, 6, 7 and 8, were obtained from baths containing 4 percent ammonium perchlorate, 0.5 percent peptone, 1 to 2 percent free lactic acid and 8, 6, 4 and 2 percent lead as lactate respectively. This series shows that a high concentration of lead is not necessary. A comparison of these two series shows the importance of a little free lactic acid.

shown in Fig. 1. Similar experiments, except without free lactic acid, gave good deposits only with the higher lead content.

The best deposits were obtained by the use of low current densities. The maximum current density that gave good deposits from these solutions was about 0.4 amp. per sq. dm. (3.6 amp. per sq. ft.). When a current of 0.6 amp. per sq. dm. (5.4 amp. per sq. ft.) was used, the edges of the cathode were very rough.

Substitution of Other Salts for Ammonium Perchlorate.

If the ammonium perchlorate exercised a beneficial action merely because it is a strongly dissociated salt of a strong acid, any other similar salt should give the same results. With sodium naphthalene sulfonate ($C_{10}H_7SO_3Na$) almost as good deposits were obtained as with the ammonium perchlorate. Ammonium fluoborate was found to be less satisfactory than the ammonium perchlorate. The presence of free acid was necessary in the case of the bath containing the ammonium fluoborate, which is unstable except in acid solutions. Sodium perchlorate was found to be fully as good as ammonium perchlorate. Sodium ethyl sulfate, fluosilicic acid and potassium perchlorate were found to be distinctly inferior to the ammonium perchlorate. The use of glue in the bath containing the fluosilicic acid did not produce a good deposit. Calcium lactate gave a poor deposit. Four percent of the above salts was used in each case.

Extracts from Wood as Addition Substances.—An extract of yellow poplar wood *(Liriodendron Tulipefera, L.)* was prepared by boiling the sawdust (5 g.) in 20 cc. of a solution of 50 percent lactic acid for half an hour. This extract was added to a bath containing 4 per cent of lead as lactate and 4 percent of ammonium perchlorate. The precipitate was filtered out and the solution electrolyzed. A thick, firm and finely crystalline deposit was obtained, which was very much better than that obtained by the use of peptone. If the extract of poplar wood was prepared by boiling the sawdust in a 5 percent solution of lactic acid (100 cc.) the deposit showed peculiar inequalities, as shown in Fig. 2.

Current Efficiency.—The current efficiency was determined on a bath (200 cc.) containing 4 percent of ammonium perchlorate, 4 percent of lead as lactate and 0.5 percent of peptone. The

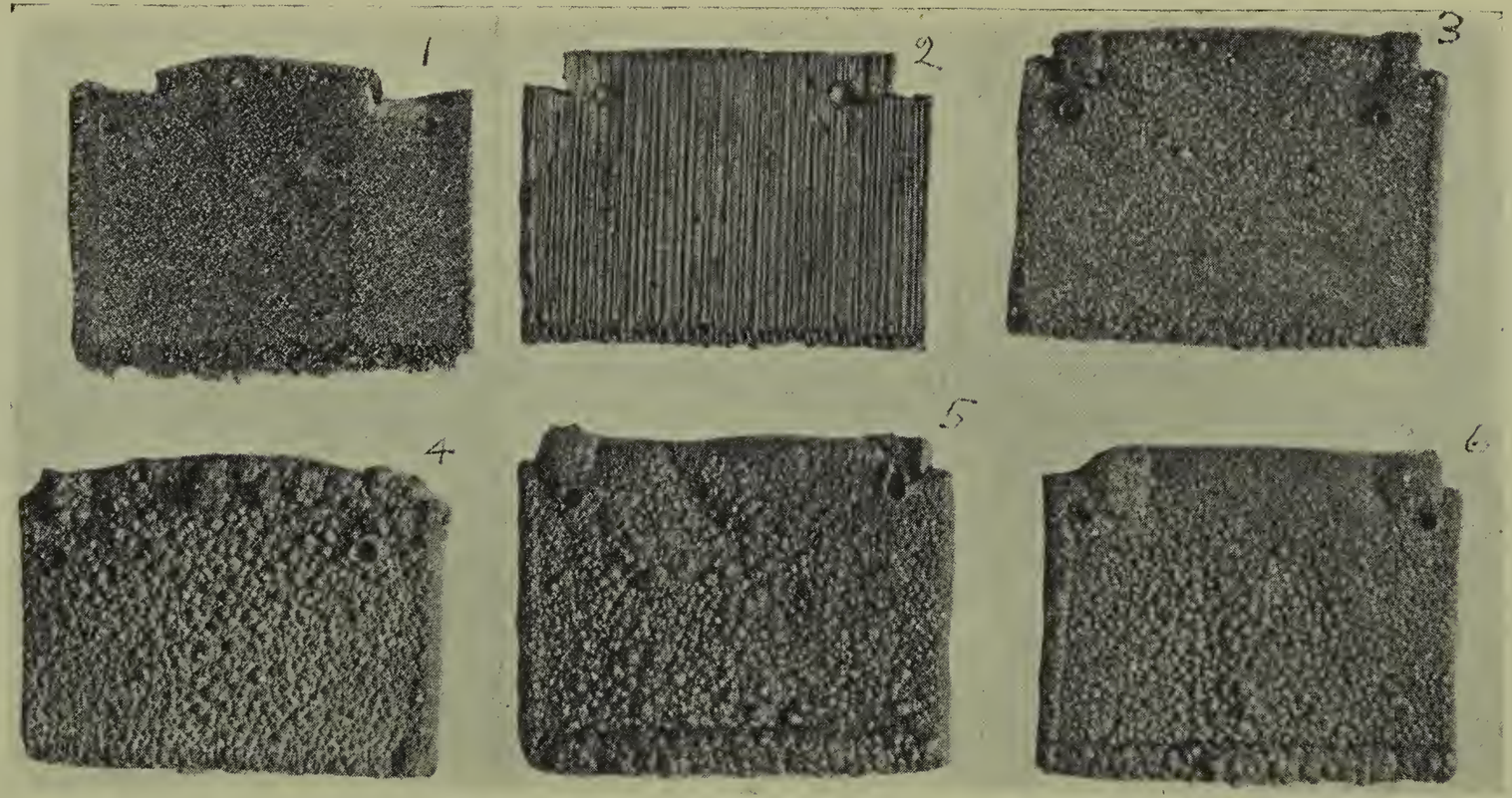

FIG. 2.—*Various Addition Agents in Lactate Baths.*

Each bath contained 4 percent lead as lactate, 4 percent ammonium perchlorate and other things as given below. The current density was 0.4 amp. per sq. dm.

1. This cathode, which is as good as any that were obtained with glue, was from a bath containing 0.2 percent glue but no free lactic acid.

2. The addition agent was prepared by boiling 2.5 percent poplar wood sawdust for two hours in 5 percent lactic acid of which enough was used so that the final bath contained 2.5 percent free acid. The peculiar striations are characteristic of deposits when the addition agents are made by boiling the wood in dilute acid.

3. This cathode differs from 2 in that the poplar wood sawdust was boiled for 0.5 hour in 50 percent acetic acid of which enough was used so that the final bath contained 5 percent free acid.

4. This cathode differs from 2 in that the sawdust was boiled in 50 percent acetic acid in place of the lactic acid and the final bath contained 5 percent free acetic acid but no lactic acid.

5. This cathode, weighing 359 g., was deposited by the use of only 0.2 g. of Barbadoes aloes. The bath contained one percent free lactic acid.

6. This cathode, weighing 311 g., was deposited by the use of 1 g. of peptone. The bath contained 5 percent free lactic acid.

cathodes were copper, and the anodes twice refined lead; a copper coulometer was run in series with the baths.

Copper Deposited in Coulometer	Theoretical Amount of Lead	Lead Deposited at Cathode	Loss at at Anode
3.7420 g.	12.2533 g.	12.1948 g.	12.2348 g.

Current Efficiency in percent at	
Anode	Cathode
99 87	99.33

The specific gravity of this deposit was determined and found to be 11.25, as compared to 11.36, which is the specific gravity of the lead gotten from perchlorate baths.[5]

The quantity of peptone required for the deposition of the lead was determined by electrolyzing a bath as long as the deposit was solid and firm, although it became rough near the end of the electrolysis. By the use of one gram of peptone, 311 gm. of lead were deposited. For depositing one ton (909 kg.) of lead, 6.4 lb. (2900 g.) of peptone would be required.

Barbadoes Aloes as an Addition Agent.—Barbadoes aloes was found to be as good an addition substance as the yellow poplar extract, and far superior to peptone, in that the deposit was less brittle and was very much smoother. Complete series of experiments upon baths containing varying amounts of lead lactate, lactic acid and ammonium perchlorate were not run, since it was assumed that the quantities of these substances for the most satisfactory results had already been determined in the experiments in which peptone was used as the addition substance. Consequently the baths that were tried contained 4 percent of lead as lactate, 4 percent of ammonium perchlorate, 1 percent of free lactic acid and 0.25 percent of Barbadoes aloes. The aloes was dissolved in water, and this aqueous solution, after the insoluble tar or gum had settled, was added to the bath. A picture of one of these cathodes is shown in Fig. 2. This cathode was left in the bath as long as a satisfactory deposit was obtained. In this way it was found that 358 g. of lead could be deposited in a solid, compact condition by the use of the water-soluble materials from 0.5 g. of the aloes. This means that 1266 g. (2.79 lb.) of aloes would be required for 909 kg. (one ton) of

[5] Trans. Amer. Electrochem. Soc., **17**, 261 (1910).

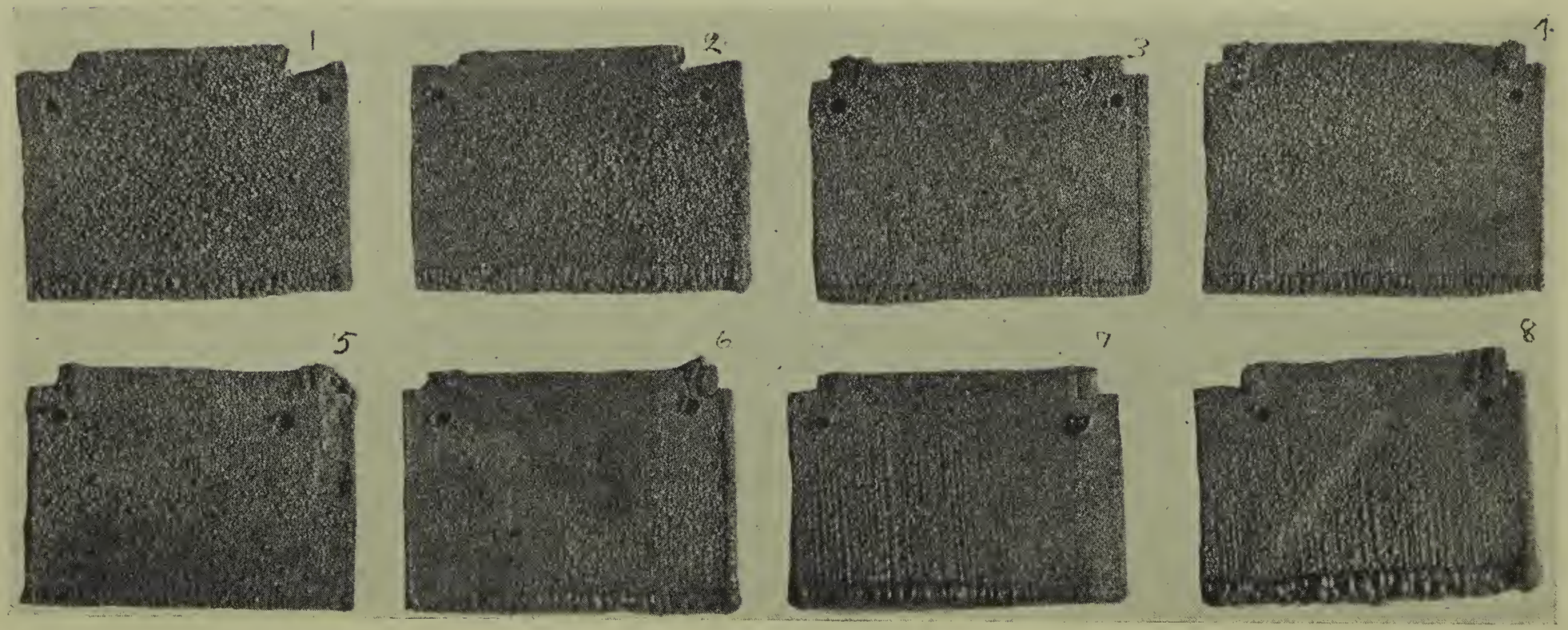

FIG. 3.—*Deposits from Lead Formate Baths.*

Cathodes 1, 2, 3 and 4 are from baths containing 2 percent lead formate, 4 percent ammonium perchlorate, 0.5 percent peptone and 10, 5, 2 and 1 percent free formic acid, respectively. The variation in the amount of free acid had very little effect upon the cathodes. Cathodes 5, 6, 7 and 8 are from baths exactly like those described above except that 0.25 percent Barbadoes aloes was used in place of the peptone as addition agent. The variation in the amount of free acid had very little effect upon the character of the deposits. A comparison of these two series shows the great superiority of Barbadoes aloes as an addition agent over the peptone.

lead deposited. A lead cathode weighing 3220 g. (7.12 lb.) was deposited from a lactate bath of the above composition; the volume of the bath was 800 cc., the cathode was 10 x 12.5 cm. (3.9 x 4.7 in.), the current density was about 0.2 amp. per sq. dm. More of the aloes was added from time to time during the electrolysis, whenever the crystalline appearance of the cathode showed that more addition substance was needed. No record of the total quantity of aloes used was kept. This cathode is shown in Fig. 4.

LEAD FORMATE BATHS

Good cathodes were much harder to obtain from these baths than in the case of the lactate. The small solubility of lead formate made it impossible to use a higher concentration than 2 percent of the salt. During electrolysis the lower part of any metal refining bath becomes more concentrated than the upper part if the bath is not stirred or circulated. This change of concentration shows its effect more quickly upon the character of the cathode deposit if the solution is already dilute, as in this case. Continuous stirring or circulating the bath would remove any trouble of this kind. In these experiments there was no agitation except occasional stirring with a glass rod, consequently the upper portions of the cathodes were not as smooth and firm as the lower parts.

No Addition Substance.—The cathode from a bath containing 2 percent of lead formate without any free formic acid was very crystalline, and soon short-circuited the bath, much as in the case of the corresponding lactate bath. The addition of varying amounts of free formic acid did not materially improve the bath. The addition of 4 percent of ammonium perchlorate caused the deposit to form in small rough crystals, which was slightly better than without the ammonium perchlorate.

Glue.—The addition of 1 percent of glue did not materially improve the character of the deposit. The glue in this case had the same action as in the corresponding lactate bath.

Clove Oil.—The addition of two to four drops of clove oil to these baths, either with or without free acid and ammonium perchlorate, seemed to be without effect. The deposits were essentially the same as without the clove oil.

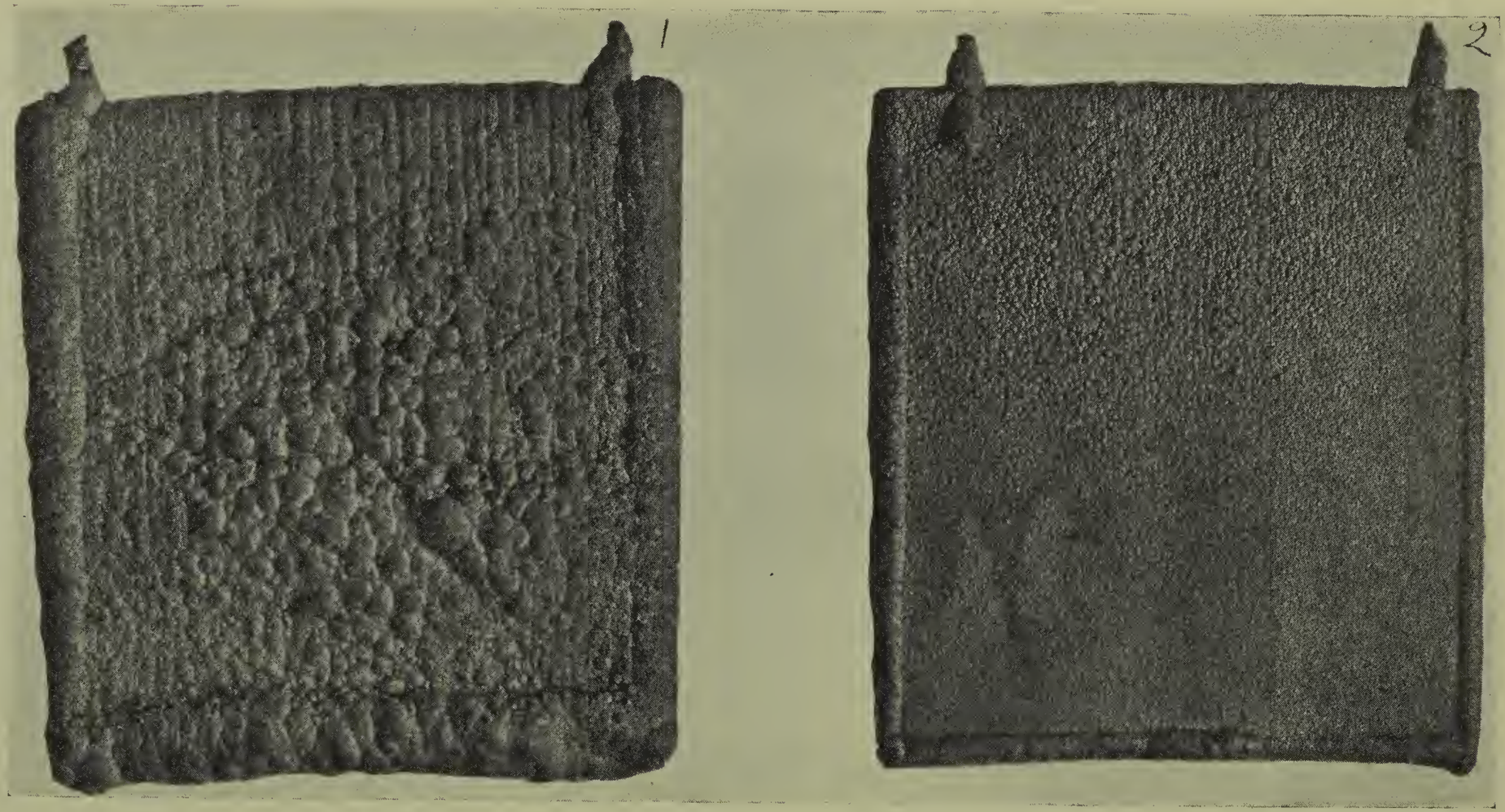

FIG. 4.

Cathode 1, weighing 3220 g. (7.12 lb.) was deposited from a lactate solution containing 4 percent lead as lactate, 4 percent ammonium perchlorate, 1 percent lactic acid and 0.25 percent Barbadoes aloes. More addition agent and more acid were added whenever required.

Cathode 2, weighing 1100 g. (2.43 lb.) was deposited from a formate solution which was made by mixing the baths from which the cathodes shown in Fig. 3 were deposited. More Barbadoes aloes and more free formic acid were added from time to time as required.

Peptone.—With solutions containing 2 percent of lead formate, 4 percent of ammonium perchlorate, no free formic acid and 0.5 percent of peptone the cathodes were free from projecting crystals, but were brittle, not firm, and the surfaces were rough. The top part of the cathodes was loose, due to the low concentration of lead. As the amount of ammonium perchlorate was decreased from 4 to 3, then to 2, and finally to 1 percent, the deposits became rougher and less firm. With 6 percent of ammonium perchlorate the deposit was rough and firm.

Effect of Free Acid.—The addition of 1 percent of free formic acid in the above bath containing 4 percent of ammonium perchlorate greatly improved the deposit, making it much smoother and firmer. An increase in free formic acid to 2 percent gave a still better cathode, while with larger amounts of free acid the deposits seemed to become more crystalline and more rough. These cathodes are shown in Fig. 3. A current density of 0.4 amp. per sq. dm. (3.6 amp. per sq. ft.) was used. Higher currents had the effect of making the deposits much rougher.

Aloes as an Addition Agent.—Peptone was not the ideal addition substance, since the deposits were not entirely smooth and firm, but were brittle and granular. Aloes was much the most satisfactory addition substance of those that were tried. In this work Barbadoes aloes was added to the complete bath, which was then warmed in order to bring the aloes into solution. After cooling the bath was filtered in order to remove the insoluble gum. Fig. 3 shows some cathodes from solutions with 2 percent of lead formate, 4 percent of ammonium perchlorate, 0.25 percent of Barbadoes aloes, and free formic acid varying in amount from 1 percent to 10 percent. As all these cathodes seemed to be about equally good, there was consequently no advantage in using more than 1 or 2 percent of free formic acid. These cathodes were very smooth and bright in color, but finely crystalline and brittle. Without the free acid the deposits were rougher and less firm, showing indications of sponginess on the upper portions where the concentration of the lead was low. Decreases in the amount of ammonium perchlorate in those baths containing no free acid produced much rougher deposits with more spongy formations near the top.

These deposits from the formate baths with the Barbadoes aloes as an addition substance are exceptionally smooth and satisfactory. The appearance of the cathodes is very similar to the best cathodes that can be obtained from any of the baths using strong acids such as the fluosilicate or perchlorate. Fig. 4 shows a large cathode which was deposited from a formate bath containing Barbadoes aloes. This bath was made by combining the baths from which the cathodes shown in Fig. 3 were plated. This cathode is one-half inch thick, and weighs 1100 g. (2.43 lb.). The current density was about 0.2 amp. per sq. dm. (1.8 amp. per sq. ft.). An unfortunate accident broke the container, and thus ended the attempt to produce a very thick cathode.

The substitution of ammonium fluoborate for ammonium perchlorate in the peptone solutions gave a cathode that was covered with projecting crystals. Sodium naphthalene sulfonate gave almost as good a cathode as when the ammonium perchlorate was used. Only one experiment was tried with each of these salts, consequently it is unsafe to draw definite conclusions.

Summary.

This paper gives the details of experiments on the conditions under which a smooth, thick, firm, crystalline but rather brittle deposit of lead can be obtained from lead lactate and lead formate baths.

The baths that gave the best deposits contained 4 percent of lead as lactate or 2 percent of lead formate, 1 to 5 percent of lactic or formic acid, 4 percent of ammonium perchlorate and 0.25 percent of Barbadoes aloes. This mixture was warmed and then filtered in order to remove the insoluble gum which was in the aloes.

A current density of 0.4 amp. per sq. dm. (3.6 amp. per sq. ft.) was found to be best; with higher current densities the deposit became rougher.

Thick deposits were obtained without difficulty. The specific gravity of this electrodeposited lead was 11.25 and 11.28 for the lactate and formate baths respectively.

Other addition agents were tried in the place of the Barbadoes aloes, but of these peptone was the only one of any marked value.

It was found that sodium naphthalene sulfonate could be used

in the place of ammonium perchlorate. Other salts of strong acids did not give as good results as the perchlorate.

The only disadvantages in the use of baths containing lead salts of weak acids are:

(*a*) A low current density must be used.

(*b*) The deposits are brittle as a result of the large amount of addition substances that must be used.

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DISCUSSION.

W. LASH MILLER: I ask whether in the literature there is any chemical investigation of the composition of aloes residue?

F. C. MATHERS: I do not know. I have seen no statement concerning the composition of this aloes residue.

F. C. FRARY: Has Dr. Mathers determined how much of his addition agent or colloid went into the cathode? It is well known that in the case of the glue process with fluosilicate a certain amount of the glue goes into the cathode and changes the properties of the lead appreciably.

What is the relative cost of ammonium perchlorate and fluosilicic acid? Hydrofluoric acid, in its commercial form, is cheap, and silica also, so that from the two fluosilicic acid salts can be made for next to nothing.

In regard to one of the deposits mentioned, where it is said that a black spongy deposit appeared soon after beginning electrolysis, possibly the difficulty was that the lead was being deposited from the bath faster than it was being dissolved. In this particular case I believe perchlorate was not added, and the deposit, good at first, rapidly became dark. It is well known that when we electrolyze acetates, acetic acid is broken up at both anode and cathode, and it would seem probable that the function of the perchlorate was to introduce a substance which would dissolve the anode properly and not be itself decomposed

by the current. This phenomenon of the formation of a spongy deposit when the solution becomes impoverished in metal is one which is quite common in the electrodeposition of copper, and might occur here also.

Did Professor Mathers make any determination of the changes of concentration of the lead in an acid solution containing perchlorate on continued electrolysis, whether the lead content was maintained constant and whether the acetate was gradually destroyed as it would have been without the perchlorate?

F. C. MATHERS: The addition substance is deposited with the lead upon the cathode. No attempt was made to determine the quantity of the occluded addition agent. Since larger amounts of the addition agents are required in these baths than in the fluosilicate or perchlorate solutions, the deposited lead shows the effect to a greater extent, being very brittle in some cases.

In large quantities sodium or potassium perchlorate can be purchased for 10 cents per pound. I do not have any price upon the ammonium perchlorate.

Many deposits seem to be good at the beginning of electrolysis but soon become bad. All defects or imperfections or crystalline growths gradually become greater and greater as the thickness of the deposit increases. This is probably what occurred in the case cited by Dr. Frary. No deposit deserves to be described as "good" until it has reached such a thickness, say 0.5 to 1 cm. (0.2 to 0.4 inch), that any defects or imperfections will be readily apparent.

There could not have been any serious impoverishment of lead in these baths since the current efficiencies were near 100 percent at both the anode and the cathode. No experiments were made to determine whether there was any decomposition of the acetate radicle at the anode during the electrolysis. This decomposition, if any, could not have been very large otherwise the current efficiency at the anode would have been noticably low.

Ammonium perchlorate or other salt was necessary for the formation of good deposits. I do not think that bad deposits in the absence of the ammonium perchlorate were due to decomposition products of the acetate migrating from the anode to the cathode. The deposits were not perfect in the beginning when

there had not been time for any solution to diffuse to the cathode. I think that the perchlorate radicle must have some influence upon the deposits since other acid radicles were less beneficial.

Jos. W. Richards: I take it for granted that the ultimate object is to find a bath which would be suitable for refining or depositing lead. In that case it would be important to get some quantitative idea of the amount of the addition substances which must be added from time to time, that is, the rate at which the addition is exhausted, because its commercial use will depend entirely on the cost of the addition agent which must be added to it.

G. A. Roush: One point of interest is that the density of the metal mentioned in every case is lower than the density of cast metal. This is interesting, in comparison with the paper by Mr. Addicks, which was presented yesterday, in which he states a fact which is already pretty well known, that electrolytic copper is almost always higher in density than worked cast copper.

Jos. W. Richards: Cast copper usually contains gases which make its specific gravity lower than that of electro-deposited copper, while the cast lead does not contain such gases.

F. C. Mathers: About 2.8 pounds of the aloes is required for a ton of lead. The crude aloes is quoted at something like ten to twenty cents a pound in the drug journals. I do not know what it would cost in large quantities. It would probably not be very expensive. This research was not so much to find a good plating and refining bath as to see what would be done with the lead salts of the weak acids. Some people said that good deposits could not be obtained from these solutions and I thought that it would be interesting to show them that it could be done.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 2, 1914, President F. A. Lidbury in the Chair.

ELECTRODEPOSITION OF TIN FROM TIN SALTS OF MINERAL ACIDS.

By Frank C. Mathers and Barrett W. Cockrum.

This paper gives the results of attempts to obtain smooth, finely crystalline deposits of tin from tin salts of mineral acids. A very extended research involving experiments with 179 various combinations of conditions and compositions of baths was made, but no entirely satisfactory deposit was obtained.

The baths contained four percent of tin as stannous chloride, fluoride, perchlorate or fluoborate or as stannic chloride, about one percent of the free acids, respectively, and 0.05 to 0.5 percent of various addition agents. Other salts, generally the ammonium salts of the acids, were tried in some of the baths. The addition agents which were tested were:—clove oil, peptone, gelatine, glue, phloridizin, Barbadoes aloes, sandalwood oil, residue from Curacao aloes, tannic acid, levulose, eucalyptus oil, catechu, benzoic acid, caffeine, pyrogallol, formin, licorice extract, oxalic acid, salicin, saponin, podophyllin, jalapin, digitalin, morphine and gum Arabic. The current density was 0.4 amp. per sq. dm. Most of the experiments were run at room temperature since a temperature of 80° C. did not materially improve the deposits.

The best deposits were obtained at room temperature from stannous fluoborate saturated with clove oil or containing 0.1 percent digitalin and from stannous perchlorate containing 0.1 percent phloridizin. These deposits were firm and free from loose trees or projections but they were very crystalline. Fig. 1 shows samples of these cathodes. The deposit from a perchlorate bath with glue is shown for comparison. It is a better cathode than was obtained from most of the other baths.

The authors feel that no combination of the salts, addition agents and conditions that were tried in this work will give a

good tin plating or refining bath. Any further work on this problem should be with new addition agents.

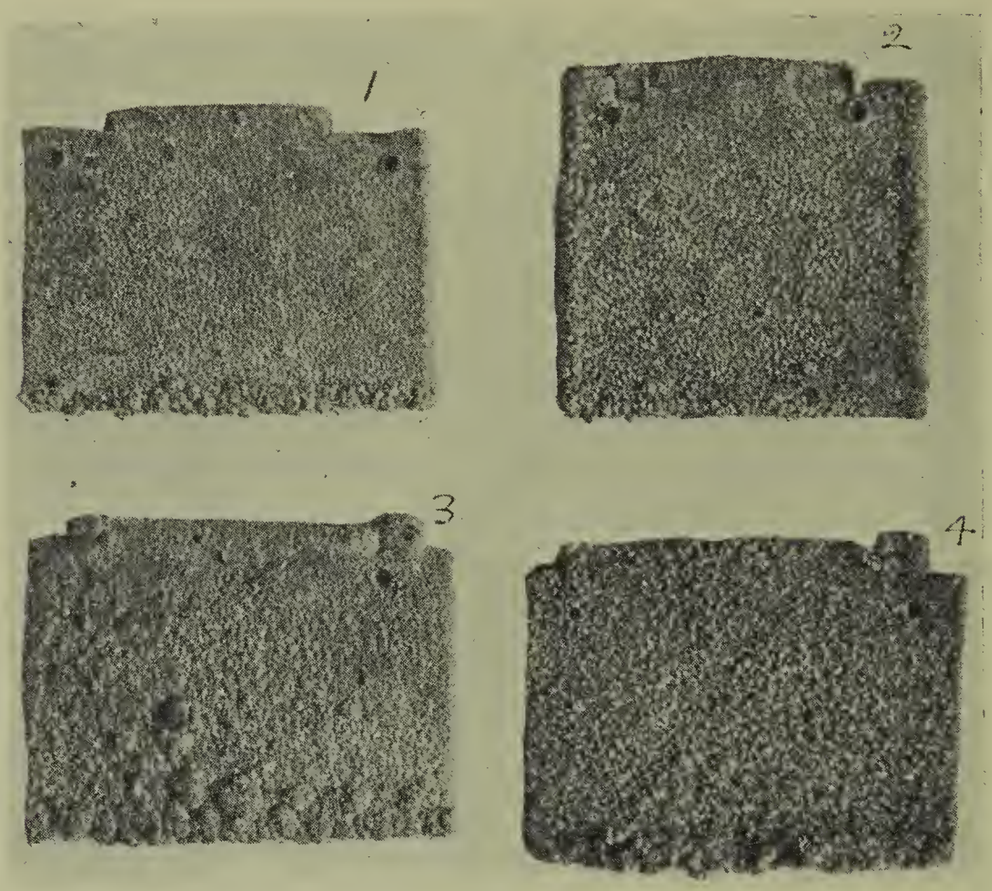

FIG. 1.

These baths contained four percent tin, about one percent of free acid and other things as given below. The current density was 0.4 amp. per sq. dm. These cathodes, except the one with glue were the best that were obtained.

1. Stannous perchlorate, 4 percent ammonium perchlorate and 0.25 percent phloridizin.
2. Stannous perchlorate and 0.25 percent digitalin.
3. Stannous fluoborate saturated with clove oil.
4. Stannous perchlorate and 0.25 percent glue.

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DISCUSSION.

H. C. CHAPIN: Were these deposits made at high or low temperatures?

F. C. MATHERS: These deposits were made at room temperature in most cases.

H. C. CHAPIN: Tin can be obtained in two modifications, the white or ordinary tin and the grey or brittle, powdery kind. I do not remember what the transition temperature is.

F. C. MATHERS: About 20°.

H. C. CHAPIN: I suppose that the temperature would have a considerable influence upon the deposit and that tin deposited at a temperature below 20° would be the grey modification.

F. C. MATHERS: These samples were deposited at room temperature which would be above the 20°. It would be interesting to try an experiment at a temperature lower than the transition point. I shall try it.

O. P. WATTS (*Communicated*): Experiments with tin perchlorate carried out by Mr. E. A. Richardson at the University of Wisconsin in 1910 gave results similar to, or possibly slightly more satisfactory than those reported in this paper. The experiments were tried in the hope of finding a bath which would yield a good deposit, and at the same time show better anode corrosion than the well-known pyrophosphate solution. A bath was prepared by electrolyzing with tin anodes a strong solution of perchloric acid, containing also some sodium chlorate and sodium chloride. The deposit obtained from this bath was of a dull grey color, and at 14 amperes per square foot became crystalline in three hours. The addition of 0.4 gram of dextrine per liter improved the deposit somewhat, and no trees or nodules formed in three hours electrolysis at 29 amperes per square foot, although the deposit developed a finely crystalline structure. An hour's deposit seemed to be the maximum for a fairly smooth plate.

F. C. MATHERS: The deposits obtained by Dr. Watts are typical of ours. In many cases the deposits seemed good for a short time, after which the crystals became more and more prominent. Addition agents had less influence in restraining the formation of crystals on tin cathodes than in the case of any other metal that I have ever tried to deposit.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 2, 1914, President F. A. Lidbury in the Chair.

THE ELECTROLYTIC INSULATION OF ALUMINUM WIRE.

By C. E. Skinner and L. W. Chubb.

Copper has always been the most largely used metal for the conduction of electrical current on account of its relatively low cost; its high conductivity; its superior mechanical characteristics and the ease with which it may be worked. For equal resistances and equal lengths of conductors, the cost of different kinds of metallic wires would be approximately proportional to the product of the specific resistance and the cost of the metal per unit volume. At present on this basis, iron and aluminum would be cheaper than copper. When the problem is that of getting the maximum conductance in a given space, silver is the only metal which is better than copper, but the cost of this metal is prohibitive in practically all applications of this nature.

The following table shows the relative cost of a number of the more common metals for equal resistances and equal lengths of conductors, also the relative cross-section for equal conductance. In both cases copper is taken as the unit:

Metal	Cost of Equal Resistance Per Unit Length	Cross-Section for Equal Conductivity
Iron	.46	6.1
Aluminium	.67	1.6
Copper	1.00	1.00
Zinc	1.05	3.6
Lead	4.30	12.0
Tin	17.90	6.0
Nickel	29.4	7.2
Silver	64.2	.95

It will be seen from the above table that where the space occupied by the conductor is not a governing factor either iron or aluminum will be cheaper than copper, and both of these metals are not infrequently used for conductors for the transmission of current on pole lines, etc.

In the construction of electrical machinery, space is usually a governing factor, and copper has been found more suitable in every way than the other metals. Copper, however, requires—for coil work—a certain amount of insulation having a definite thickness (it depending upon the conditions of application), and, consequently, in the average coil the total percentage of copper in the cross-section of the coil is reduced by the insulation requirements. In coil winding, the thickness of the insulation on individual turns does not vary greatly, as a rule, regardless of the size of wire used. Consequently, the percentage of copper to insulation decreases rapidly as the size of wire is reduced.

It is evident that any gain in the efficiency of the conducting material must, therefore, be obtained by increasing the conductivity or decreasing the thickness of the insulation. The authors have found that it is possible to produce an insulating coating on the surface of aluminum wire which is so extremely thin that on many of the smaller sizes of wires it is possible to get a higher space efficiency, so far as the conducting material is concerned, in a coil with aluminum than with copper, even when the thinnest possible insulation is applied to the copper.

The skin or thin layer of natural oxide which is so objectionable in the soldering of aluminum is a non-conductor of high dielectric strength and for several years engineers have been trying to strengthen the film of oxide so that it could take the place of the fibrous insulation ordinarily used on copper wire. Robt. Hopfelt and others in 1906 and 1907 showed that coils of bare aluminum wire could be used to wind magnet coils, solenoids, etc. The wire after being wound was soaked and then heated by passing current through the coil. This process would form an oxide which was said to withstand a potential as high as 0.5 volt, and if formed between all adjacent turns of the layers of the winding and with insulation between layers, this type of coil could be used as an economical substitute for copper coils.

Early in 1907 the authors devised a method of strengthening the coating by means of an electrolytic action which produced a coating of many times the dielectric strength of the coatings which had been obtained by former methods. It was found, however, that the successful production of an insulating coating which would withstand a potential of 500 volts and which proved to be

exceedingly thin and which was indestructible, so far as temperature is concerned, was but the first stage in a research looking to the production of commercially satisfactory coils for electrical apparatus.

It was found that in the problem, as a whole, there was involved the question of making joints in the wire itself; the question of joining leads to the aluminum wire which could be satisfactorily soldered, as in the copper; the question of winding of the coils which were found to require different treatment from the ordinary coils; the question of securing proper grades of wire; and many other details which had to be taken into account.

In the following we propose to confine ourselves mainly to those points which, we believe, would be of particular interest to the members of this Society.

It is well known that aluminum as an anode in the solutions of certain salts, polarizes or films with a coating which tends to prevent the flow of current from the electrode to the electrolyte. The polarization voltage between the electrolyte and aluminum builds up to a certain critical value, dependent upon the salt dissolved, the density, temperature, former history of the solution, etc.

Several explanations of the nature and cause of this action have been put forth at various times. One theory assumes it to be a counter e.m.f. produced by charge similar to the action in the storage battery. Another theory states that a gas film is formed between the metal and the oxide film which is under high pressure because of the electrostatic attraction and therefore of high dielectric strength. The action of the film, however, seems to indicate a film of alumina which is very thin, of high dielectric strength, and high specific capacitance. Nascent oxygen formed by the passage of current from any hole or local weak area of the oxide film would oxidize more metal at that point and increase the thickness of the insoluble compound. In this way the film is built to a uniform strength all over the surface of the anode.

After such a film is formed the anode may be withdrawn from the electrolyte, washed, and when dry will be found to have a dielectric strength approximately equal to the voltage at which it was formed. This film after drying is the same as the insulation now used on the bare aluminum wire. The development

of the process of insulating wire in this way went through several stages, some of which will be covered briefly.

Process of Insulating.—Coils wound and then treated by water and heat were found to be full of short circuits unless wound rather loosely and carefully. So from the first it was thought that the wire should be insulated before winding.

The first process of insulating by electrolysis consisted in winding the wire into skeins, dipping them into the electrolyte, and after treatment and subsequent washing, the wire was wound into finished coils. This method was rather expensive because of the additional handling, rewinding and the great amount of power required for treatment. To prevent arcing and consequent roughening of the wire, the current had to be fed into the skein through the two ends. The area of fresh wire presented to the

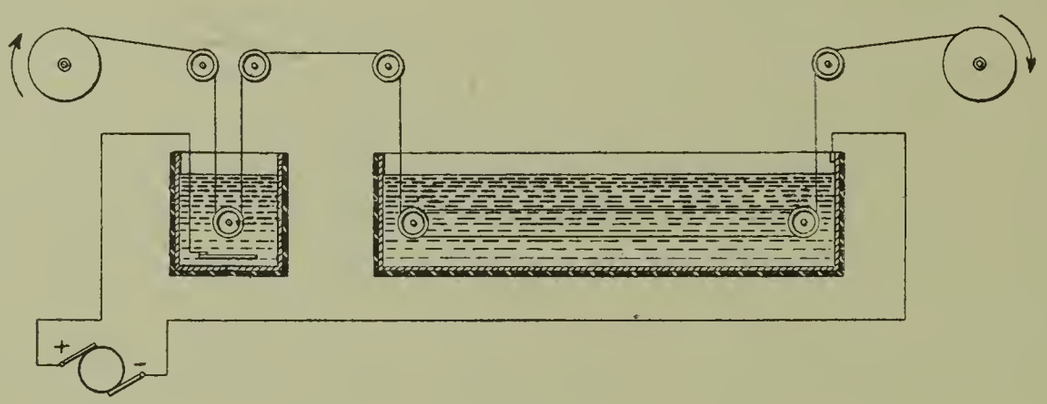

FIG. 1.

electrolyte was large and a high current density could not be used because the leads of the skein would be fused with the high current. The film therefore had to be formed slowly with an increased expenditure of energy and time. In the subsequent development continuous methods were employed in which the wire as an anode was drawn through a bath of electrolyte. In the first continuous process the wire was drawn into and out of the bath over wooden sheaves and the current fed into the wire with a sliding or rolling brush at the entering end. The greatest difficulty with this arrangement was that the contact which fed the current to the wire was continually sparking, which roughened and burnt the surface. Also, the current being fed into the wire from the untreated end gave the highest voltage between wire and electrolyte at the point of first contact. If the current could have been fed in from the outgoing end the resistance drop

through the wire would have lowered the potential between electrolyte and wire at the incoming end. The next improvement upon this was to first lead the wire as a cathode through an additional bath (Fig. 1). In this case the commutation of the current was satisfactory but the high voltage at first contact still gave trouble. The travel of the wire was reversed in some tests which gave a drop through the wire and increasing voltage during the passage through the bath. This, however, made the wire pass through the last bath as a cathode after it had been insulated.

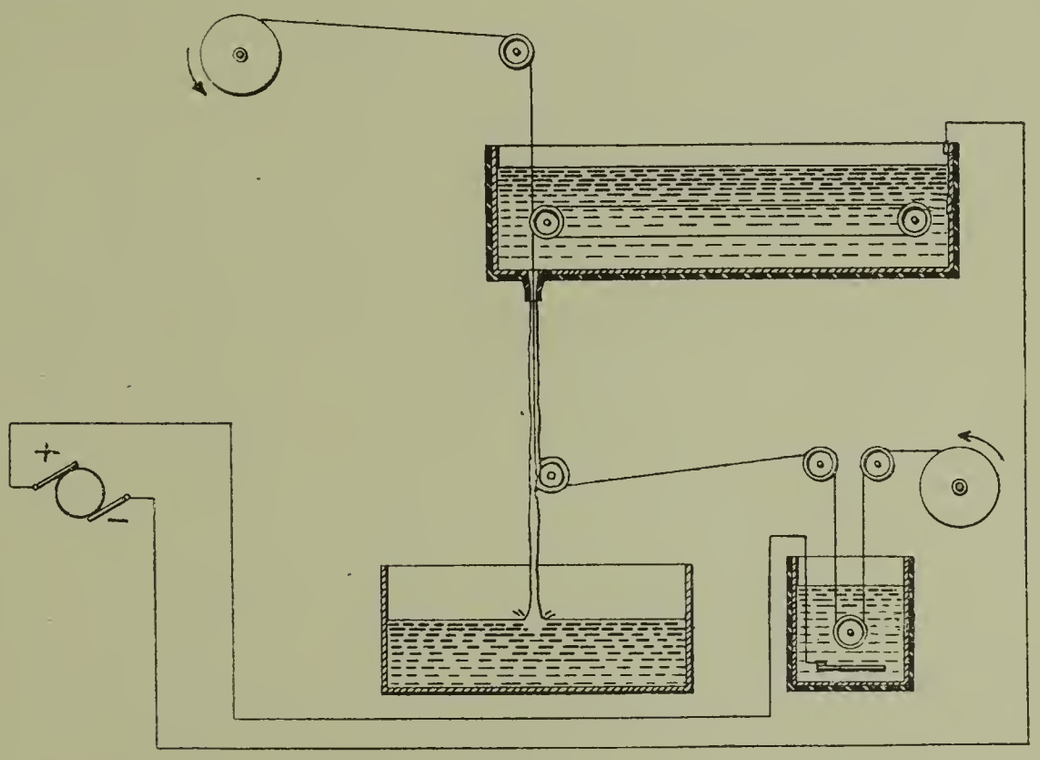

FIG. 2.

In the third method the voltage between wire and solution at the first point of contact was lowered by having a resistance drop in a column of electrolyte flowing from the bottom of the treating tank (Fig. 2). Another method consisted in drawing the wire several times around a large laminated core in which an alternating flux was maintained. By this method two wires entered at low voltage and the potential increased with each turn around the core.

In all of the above continuous methods of treating, the wire was hardened by the passage around so many sheaves so that a straight through method as shown in Fig. 3 was tried and found to give very satisfactory results. Five tanks arranged in a

straight line were used. The first and last contained hot water for washing and the middle three electrolyte. The wire passed from tank to tank through glass tubes and the solutions were kept apart by blowing air through a tube sealed to the center of the connecting tubes. In this method there was again no column of electrolyte at the point of entrance and it was at first necessary to form the coating in two steps, of 200 and 400 volts respectively. This was superseded by the present method in which the wire also goes straight through and the increase in voltage is again established through a small weir and spout through which the wire enters. This latest apparatus, a detail of which is shown in Fig. 4, has the additional advantage of allowing the removal and starting of the wire without threading through.

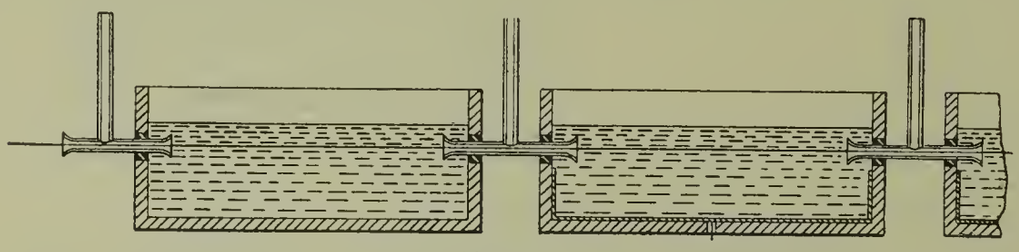

FIG. 3.

Electrolytes.—Several electrolytes were used and they gave quite different results. The principal differences were the amounts of current and energy required to bring the wire up to voltage and the possible speed with which the wire could be run through the bath. Solutions of borax, ammonium borate and sodium silicate were the best, the last being the best of the three.

Speed of Treating and Energy Required.—It was found that less energy was required per unit of surface if a high current density was used, and the voltage brought up quickly. The speed and current density were limited in the case of small wires by the fusing of the wire between the tanks, and with the large wires by the limited capacity of the generator used in the experimental work. The speed of treating wire varied from 40 to 150 feet per minute for the different sizes of wire used and the consumption of energy was from 0.15 to 0.80 watt hours per square inch of surface treated.

Properties of the Coating.—The insulation formed by the above processes is a thin film of alumina which is smooth and iridescent or abrasive and white. The smooth films form with a greater

expenditure of energy and in inferior electrolytes. The white film formed in sodium silicate at about 425 volts is rather smooth to the touch but so abrasive that it cuts and grinds the hand while coil winding. In some cases this treated wire running through the hand of the winder has cut through the skin and into the flesh before discovery.

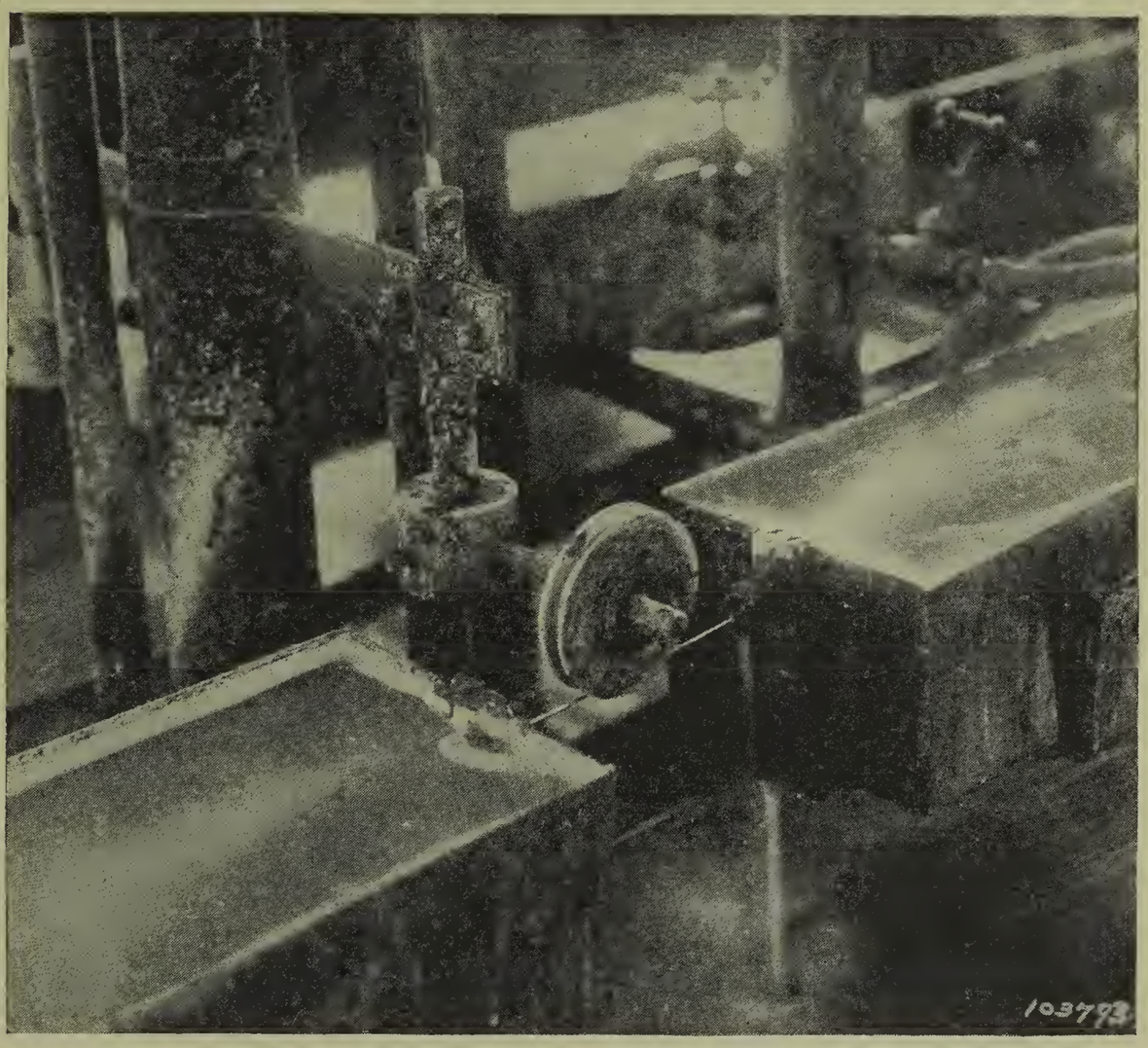

FIG. 4.

Wire as cathode passing through left-hand tank, then through right-hand tank, where it is treated as anode. Light streaks in right-hand tank are diffracted images of luminescent wire anode.

The dielectric strength is far superior to the oxide coat formed by the former methods, for instead of standing a maximum of 0.5 volt two wires electrolytically treated and twisted tightly together without abrasion will stand from 200 to 500 volts.

The thickness of the film is apparently variable. Tests of change in diameter of wire before and after treating and tests

of reduction in metal section by resistance measurements before and after treating show the coating to be from 0.0001 to 0.0004 inch (0.00025 to 0.001 cm.) thick.

On account of the thinness of the film it is quite flexible and does not open or appreciably weaken by sharp bends and kinks in the wire. Soft wire can be stretched 30 percent of its length without apparently injuring the insulation. Two wires at a potential difference of 250 volts can be crossed and pressed together at the intersection until the wires are dented to such an extent that the intersection is no thicker than a single wire. If there is no relative motion between the surfaces in contact the insulation will not break down but the slightest movement of one of the wires will cause a grinding between the surfaces which will at once cause a short circuit.

The insulating coating is fire-proof and has sufficient strength mechanically to support the wire in small coils when the metal is above the melting point. Small coils run with the wire molten will fall to pieces if the current is suddenly stopped, but if the current is gradually lowered the metal will solidify and remain intact.

Application of the Wire.—Electrolytically insulated aluminum wire is suitable for many classes of work requiring wires of relatively small size where the insulation requirements are not excessive and particularly where temperature requirements are severe. The very small sizes, No. 30 B & S Gauge and smaller, cannot be satisfactorily used on account of the difficulty in drawing the wire and in subsequent handling. In the very large sizes there is no advantage in the use of aluminum wire where the space is restricted. This wire has been found particularly advantageous for use in coils which require light weight and which are subjected to relatively high temperatures, provided the insulation requirements on the individual wires are not excessive. The weight of an aluminum wire coil is from 40 to 50 percent of that of the equivalent copper coil. The thermal capacities of the two types of coils are practically the same and the temperature co-efficients are equal. The heat dissipation through the aluminum coil is found to be slightly better than the equivalent copper coil.

While this new wire has limitations, it has a decided sphere

of usefulness, which will probably be greatly extended, particularly in the event of the ratio between the base prices of aluminum to copper being decreased.

DISCUSSION.

W. R. MOTT: About seven years ago I was rather interested in the aluminum anode properties in various solutions, and at that time I conceived the idea of coating the aluminum coiled wire with the electrolytic film and filed an application for a patent in the United States Patent Office. The application was filed a few months prior to the filing of the Westinghouse application. We lost out in the interference owing to the fact that the Westinghouse Company had reached a practical stage in the development of the process before we filed our application.

Speaking of this sodium silicate coating, away back in the history of the art, some sixty years ago, Becquerel worked with sodium silicate at fairly high voltages to form films on aluminum.

The problem of measuring the thickness of these films is very difficult.

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H. M. HALL: This paper has been particularly interesting to me since I recently conducted a few experiments along similar lines, having occasion to produce an insulating coating on square aluminum wire. As you can understand, the use of a process of this kind is more of a problem in the case of square wire than it is in the case of round wire, on account of the fact that it is more difficult to get a coating on the sharp edges which will maintain itself and not break down, due either to mechanical abrasion or a little extra high voltage. I would ask Mr. Skinner if he has ever tried this process on square wires. I found that I had to draw the wire with edges rather blunt. It was still square but the edges were considerably rounded.

Has Mr. Skinner tried this process with alternating current instead of direct current? With alternating current at 25 cycles and a current from 25 to 50 amperes on a No. 16 square wire, using a sodium silicate solution, we were able to get very satisfactory results, and produce a highly insulating, desirable coating. I believe these coatings, however, are very largely aluminum silicate, in the case of the sodium silicate electrolyte; there is also probably a good deal of alumina present.

From Mr. Skinner's paper I gather that the wires being coated carry such a high current that they are appreciably heated. If that is the case would it not be likely to be injurious if the wire is furnished on certain definite specifications and is heated enough during the process to change the properties and strength of the wire?

C. E. SKINNER: Ordinarily we use direct current because of the better control and higher treating efficiency, so that these figures refer to direct current.

We have done some coating of square wires, but it is a fundamental principle in electrical work not to use wire having square corners for windings. We must have round corners or we get into other troubles, so we have not experienced difficulty, as we do not coat wires having sharp corners.

The question of the use of alternating current for coating wire is covered in the paper (page 141).

The physical properties of the wire after treatment have not been studied for the reason that for the use we have been making of the wire, we have required practically dead soft wire to start with. We want the soft wire for easy winding and for high conductivity. Whether or not the heat is sufficient to affect seriously the physical properties of hard wire we do not know. With some of the electrolytes, the temperature of formation is lower and the process could probably be applied without affecting hard drawn aluminum wire.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 2, 1914, President F. A. Lidbury in the Chair.

ELECTRO-PERCUSSIVE WELDING.

By C. E. Skinner and L. W. Chubb.

Introduction.—The soldering and welding of aluminum has always given trouble and the satisfactory joining of small wires has been very difficult if not impossible.

In the last few years, the substitution of aluminum wire for copper wire has demanded a good means of joining aluminum and the electro-percussive method of welding was developed primarily for this purpose after a very thorough investigation into the available methods of soldering and welding of aluminum. Several other special applications of the method are now made and the future field of application seems sufficiently promising to justify a description of this new process, which will weld practically any two metals whether alike, or different, of high or low melting point, or of unequal thermal conductance.

Welding Aluminum.—With aluminum the natural oxide, which always covers the surfaces of the two pieces to be welded, prevents the metal from flowing together after the ends have been melted in the usual way. Large wires and rods of aluminum can be welded by melting the metal under the oxide film and then suddenly pushing the ends of the two pieces together. This breaks the oxide films, allows the clean metal to flow together, and at the same time washes away the broken pieces of oxide in a surplus of molten metal. Small wires cannot be joined satisfactorily by this method, or, in air by any other prolonged heating method, because of the skill required and also because such joints in small wires, if made, are oxidized to such an extent that they are brittle and weak.

Electro-Percussive Welding.—In 1905, while experimenting with electrolytic condensers and rectifiers, one of the authors noticed that wires could be connected to the aluminum plates by

the condenser spark when the cells were discharged. It was noticed that copper wires could be attached to aluminum or that two pieces of aluminum could be joined together by the condenser spark. This was of course only a feeble welding of the metals for temporary connections. But later on, while working with aluminum wire, the demand for a welding method and a

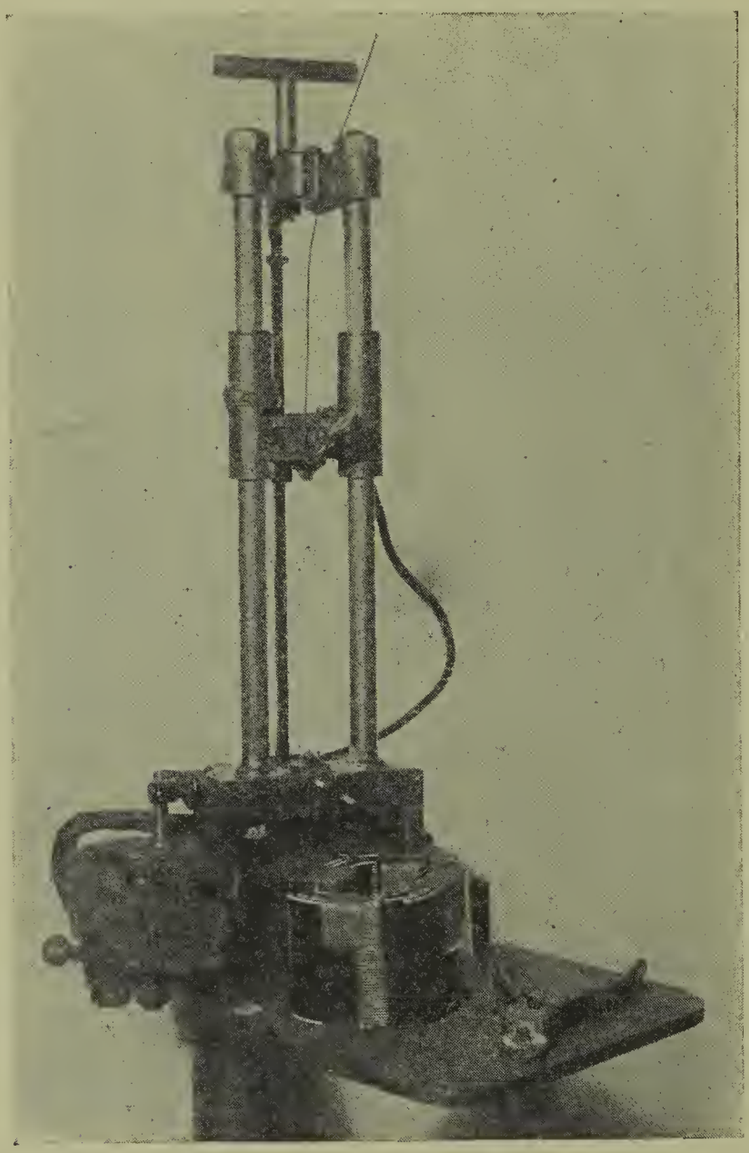

FIG. 1.

careful theoretical consideration of what happened in the early tests with electrolytic condensers made it appear worth while to try out the method of welding on a larger scale with a condenser discharge.

From the first, the same principle of a simultaneous condenser discharge and percussive engagement has been used, but during the tests and development of welding tools it was found that the

best results depend upon several variables, such as the condenser capacitance, the velocity and force of impact, the voltage, and the resistance and inductance in circuit. The first apparatus consisted of two hinged arms with wire grips in their ends. Wires placed in the grips were connected to the terminals of a charged electrolytic condenser. These arms, upon release, were drawn together with a rubber band and, at the instant of contact, the explosive condenser discharge and the forge would weld the ends together. This apparatus was not very satisfactory as it did not allow separate study of the effect of variation of velocity, momentum, kinetic energy, etc. So a second apparatus was built similar to a pile driver, with one stationary and one moving wire grip. With this arrangement the forge and velocity could be varied independently by the separate adjustment of the drop and mass of the moving part. Other welding tools have been designed in which springs have been used to shoot the wire grips together horizontally; however, these have not been as satisfactory as the gravitational type.

Process of Welding.—Fig. 1 shows a portable tool of the gravitational type set to attach a copper lead wire to a coil of aluminum wire. The welding circuit (Fig. 2) consists of a direct current line, or a generator (G) which charges an electrolytic condenser (C) through a high resistance (R). Ajustment of the voltage of the charged condenser is obtained by field control of the generator or by varying the resistors R and R'. The wires (WW) to be welded are secured in the wire grips of the welding tool which are connected to the terminals of the condenser through an inductance (L) of from 2 to 10 turns of cable. A spring switch (S) with carbon contacts, normally held closed, is connected across the jaws of the welder so that their potential difference will be zero while the wires are being put in or the finished product is being removed. The welder is connected to the auxiliary apparatus by means of long flexible cables which allow welding within a radius of about 50 feet without moving the condenser and other heavy apparatus.

The process of welding is as follows: The wires are secured in the wire grips and the ends cut off as short as possible with a suitable pair of cutters. The switch (S) is then opened which charges the condenser to the proper voltage, a catch is then

released which lets the sliding member fall and bring the ends of the two wires into percussive engagement. At the instant of contact, the short circuit current of the condenser builds up to such a value that the ends of the wire are melted by the explosive discharge and instantly forged together by the blow of the falling mass. The weld is then complete and after being removed from the machine will be found to have the strength of the original wire.

The generation of the heat is so localized, so sudden and so intense, that there is no time for unequal heat conduction through the shanks of the wire and the ends will be melted and even

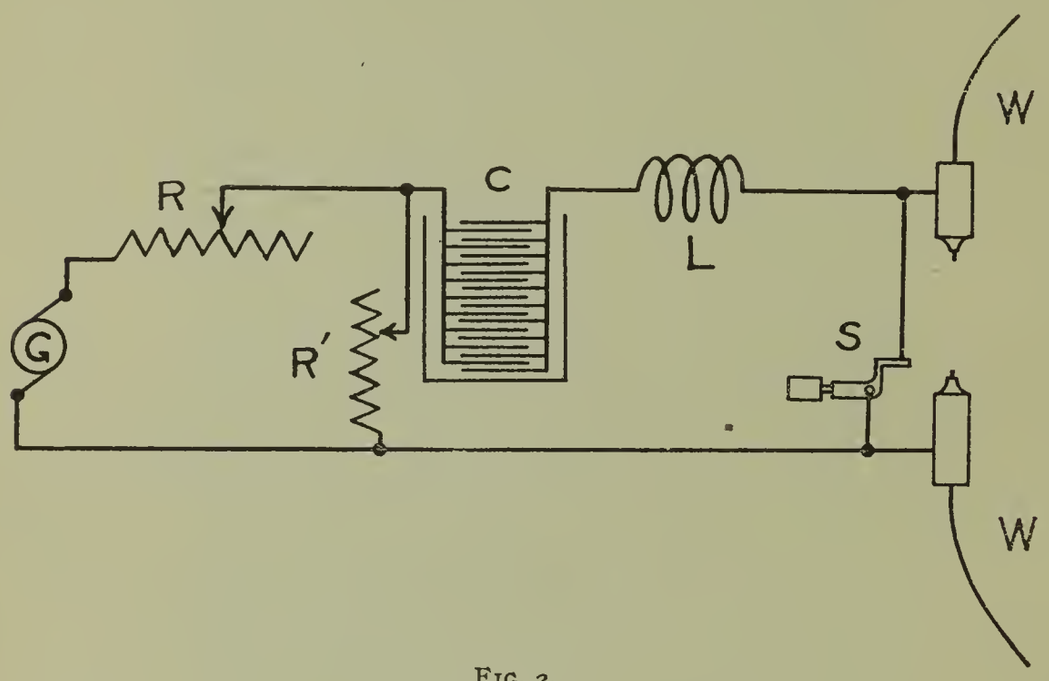

FIG. 2.

vaporized whether the melting point is high or low. For this reason metals of different kinds can be welded together independent of their electrical resistance, melting point or heat conductance. Any combination of metals which has ever been tried will weld together, but the joints will not be permanent with such combinations as aluminum and tin, or lead and iron.

The generation of heat at the point of contact depends upon having an appreciable resistance offered to the current between the wire ends, and it is therefore necessary to cut the ends of the wires in such a way that they will make a point contact preferably at the center, so that the energy of the condenser will vaporize the small section of metal and wet the rapidly approach-

ing surfaces with the intense heat of the arc. To do this the wires are cut with ordinary cutters which give a chisel edge. The two wires are cut off in such a way that the two chisel edges are at right angles and the point of first contact will be at the intersection at the center.

The time between the first contact and the finished weld is of such short duration that the exact action cannot be recorded but is supposed to be about as shown in Fig. 3. In (*a*) the wires are approaching at a velocity of from 65 to 200 cm. per second. (*b*) shows the first contact at which time the current begins to build up and heat the small section of metal carrying the current. (*c*) shows the wire ends again separated, not by appreciably retarding or reversing the motion of the upper wire but by melting and vaporizing the metal which first touched together. (*d*) shows the wire chucks closer together but the arc still burning between the wires. At (*e*) the second contact has been made, the arc extinguished and the forge of the metal has begun. (*f*) shows the completed weld after the upper chuck has come to rest and the forge is complete.

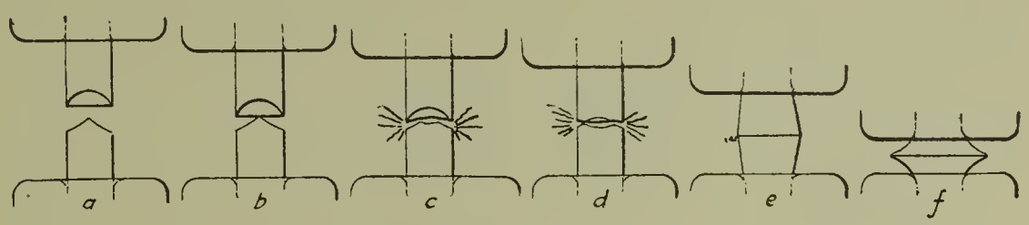

FIG. 3.

The curves of Fig. 4, drawn to scale, show the current through and the voltage across the weld during the operation of welding a No. 18 B & S gauge wire. These curves were taken directly from an oscillogram, and the power or product curve has been figured and drawn in. Electrically the weld is complete in 0.0012 second and although 23 kilowatts are being dissipated between the ends of the wire at a certain instant, the total energy used at the weld is only 0.00000123 kilowatt hour or enough to light an ordinary 50 watt, 16 candle power lamp for 0.09 second. The cost of this amount of energy at 10 cents per kilowatt hour would be 12 millionths of a cent. The letters *a, b, c, d, e, f,* have been placed along the time axis of Fig. 4 to approximately indicate the time corresponding to the various views in Fig. 3.

It will be noticed that the watt curve is oscillatory and that the negative values would indicate a return of stored energy. Such a thing would be impossible from a metallic arc but can be explained by saying that the voltage was measured above and below instead of between the wire chucks, and the storage and return of energy is from the magnetic flux produced in the steel chucks set up by the current of 500 amperes flowing through them.

The electrolytic condenser has high absorption if the current through it is reversed in an attempt to change its polarity. For

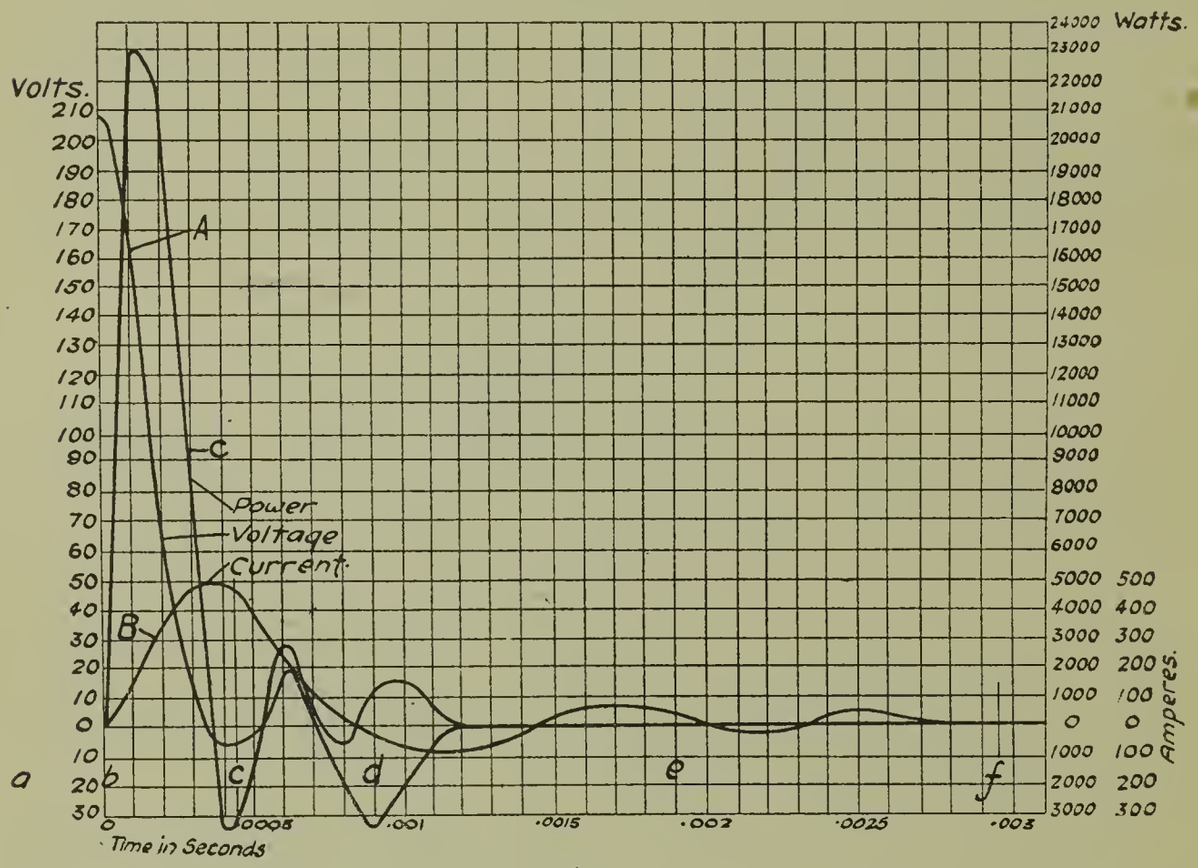

FIG. 4.

this reason any tendency toward an oscillatory discharge will be quickly damped by the internal losses of the condenser and the energy of the charge will be partly lost.

Enough resistance cannot be maintained at the weld to sufficiently prolong the current and heating until the final contact, and the very high frequency oscillation due to the small inductance of the circuit will cause an explosive snap at the weld, the current will suddenly be damped by the absorption of the condenser, the arc will go out before final contact, and the wires will not be hot enough to form a good weld. In order to maintain

the arc until final contact it is therefore necessary to lower the frequency of the discharge, and prevent the sudden rise of current at the first instant of contact by inserting additional inductance in the discharge circuit. This inductance prevents the explosive and wasteful discharge, prolongs the current, and apparently maintains the heating until final contact is effected. The inductance will of course increase the tendency toward oscillation and although the total energy dissipated at the weld may be diminished, it is more efficiently used and not wasted in noise or thrown out in metallic vapor.

The equations expressing this interesting transient are not as valuable as the ear in telling what adjustments are best for any particular weld. The mass and drop of the falling chuck must be sufficient to slightly forge the wire and then all that is necessary is to change the voltage and inductance so that the discharge sounds like a splash or thud instead of a sharp crack. Generally the operator can guess at the settings and no trials need be made. When the same work is to be repeated the machine once set will make a perfect weld every time, and the rate of welding will be limited only by the mechanical handling of the product.

The new process has some distinct advantages over the usual methods of working metals and is especially adopted to welding small sections of like or unlike metals. In many cases a saving of expensive material can be effected, in other cases work can be done which cannot be done by other methods; joints can be made without overheating the metals, and the welds will be ductile and strong.

Properties of the Weld.—Many of the alloys of most metals are very hard and brittle. As an example there are alloys near both ends of the copper-aluminum series which are unworkable, and yet electro-percussive welds between these two metals are so ductile that they may be worked in a die, forged, or rolled into thin foil. Any alloy formed at the weld must range from 100 percent copper on one side to 100 percent aluminum on the other; but possibly the brittle combinations are so thin that the joint as a whole is flexible and ductile. This joint between aluminum and copper is of great importance, as copper lead wires, which solder and connect easily, can readily be attached to aluminum coils. At first it was feared that a diffusion of the two metals

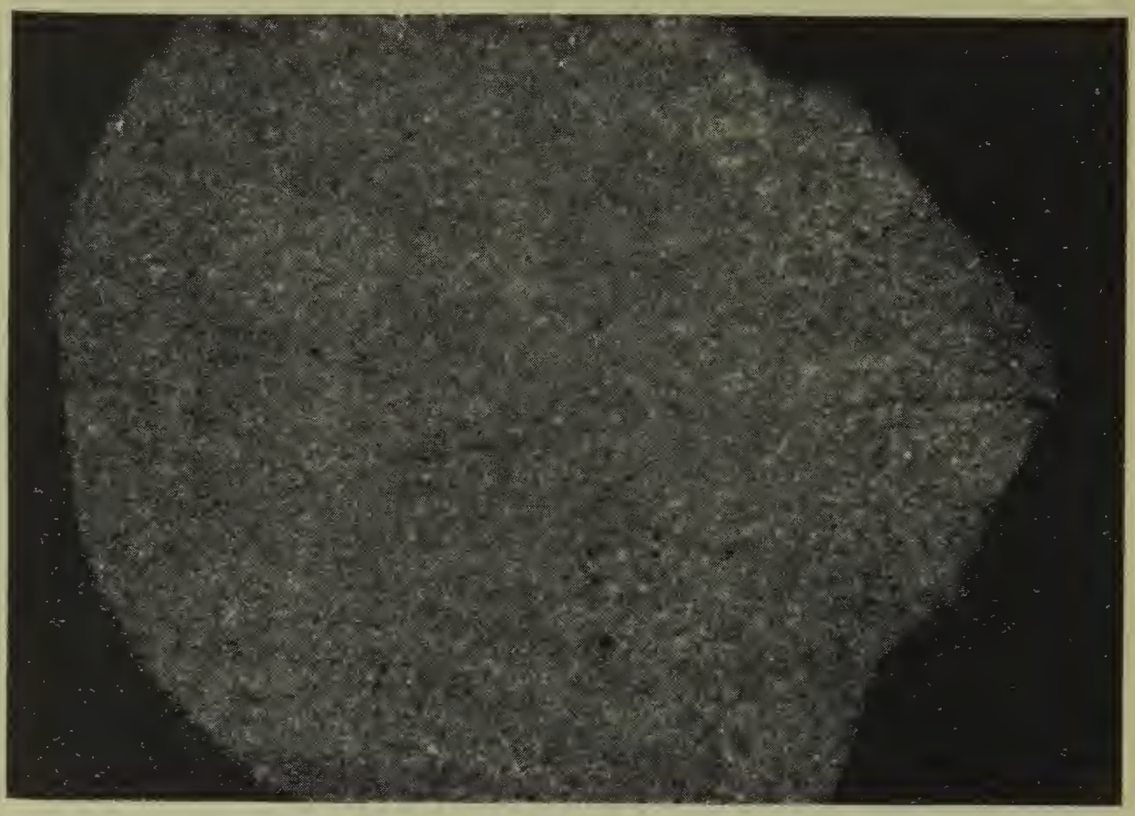

FIG. 5. Aluminum Wire. × 50

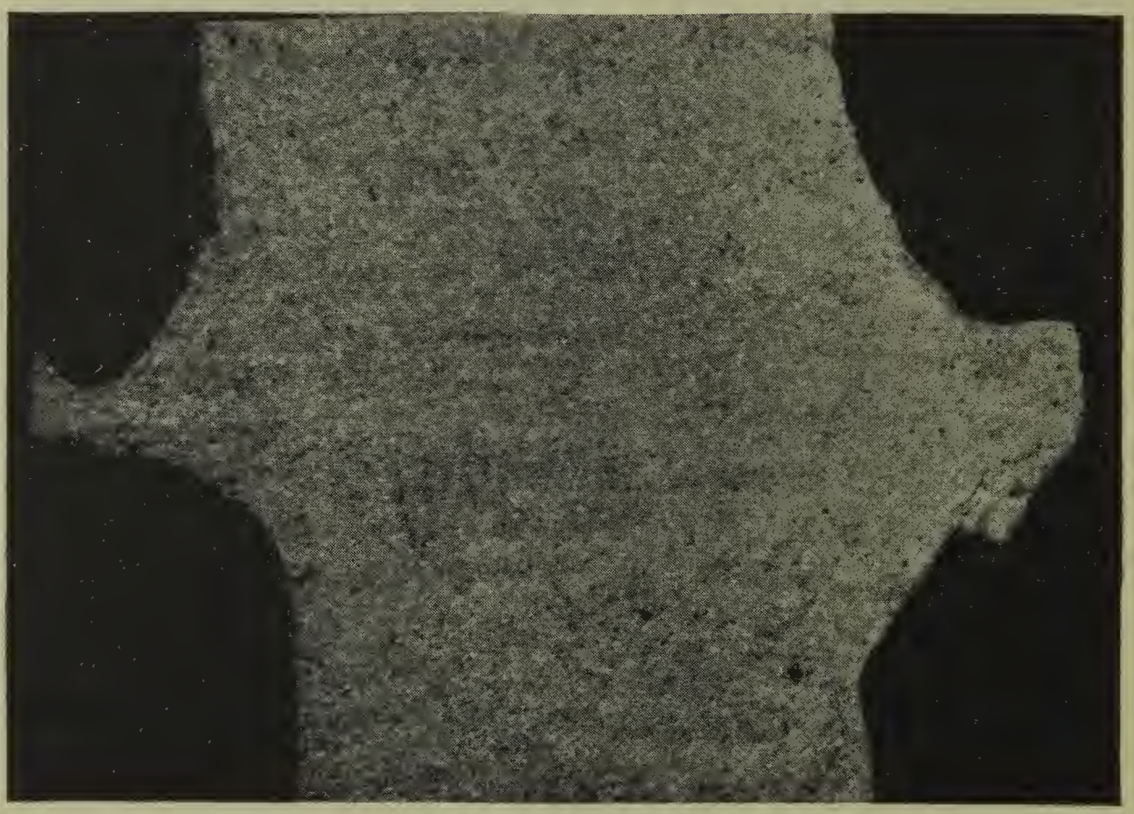

FIG. 6. Aluminum Wire. × 50

in service would finally result in a brittle joint, but tests show that after four years the joints are practically as strong and ductile as when first made. Similar ductility has been noted in almost every combination of metals when first welded, but diffusion, disintegration and loss of ductility eventually result in such welds as silver to tin, or aluminum to tin.

Metals which are either hardened or softened with heating and sudden cooling can be welded together without appreciable change in the physical properties of the material. Tempered spring steel welded, reduced to uniform diameter, and tested has shown equal or greater strength at and near the weld without any noticable change in temper. Metals such as hard drawn copper, silver, aluminum, etc., which soften with heat can be welded together without causing any local annealing, and these metals and steel when soft can be welded together without detrimental local hardening.

Several possible explanations of the constancy of the mechanical properties before and after welding can be given. First, such sudden heating and cooling may not allow change in molecular structure. Second, with hard steel, the heated metal at the weld is so suddenly cooled by conduction of heat into the two shanks of metal that it is again hardened. Third, with hard copper, silver, aluminum, etc., the heating and sudden cooling would ordinarily soften the metal, but the cold forge of the blow in welding possibly hardens it again. Fourth, the metal subjected to the sudden heat cycle may be hardened or annealed (depending upon the characteristics of the material welded), but the amount of material affected may be too small to be detected. As an example, assume a No. 18 hard drawn aluminum wire to be welded together. 0.00123 watt hour or 1.06 small calories are dissipated at the weld. Assume none of the energy lost in noise, radiant heat or metallic vapor, and one-half of the total propagated in a heat wave in each direction along the wire. It can be shown mathematically that an annealing temperature will not be reached more than 0.05 mm. from the weld. The total amount of metal softened would then be a cylinder 0.1 mm. long and 1.02 mm. in diameter. A soft insertion of such proportions could hardly be detected. Fig. 5, showing an enlarged section of such a weld, indicates that the disc of changed metal is only about

0.01 mm. instead of 0.1 mm. thick and physical tests fail to show any weakness or softness at the weld after being reduced to uniform diameter by removing the surplus metal of the burr. Fig. 6 shows a recent micrograph of a similar weld made three years ago, and it will be noted that the line of the weld does not appear and either never existed or has disappeared in the meantime.

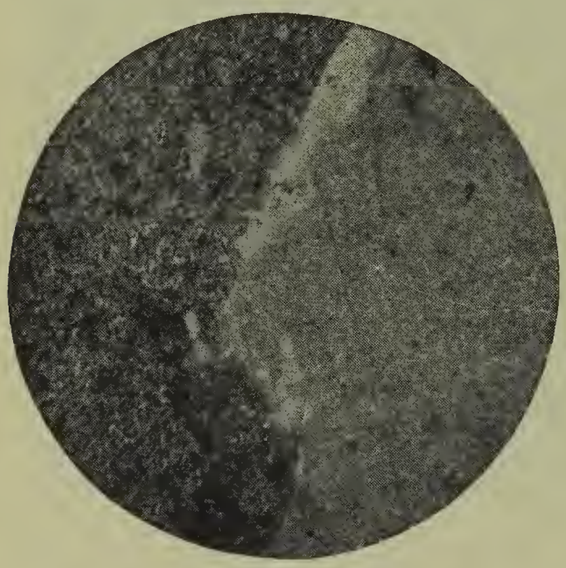

FIG. 7. Al-Cu. × 1000

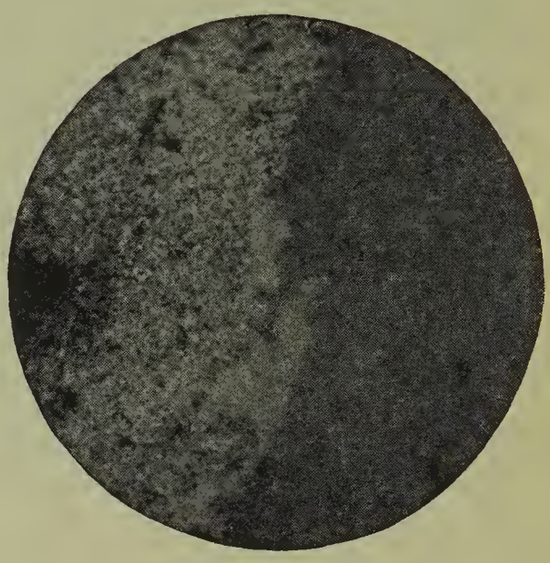

FIG. 8. Al-Cu. × 1000

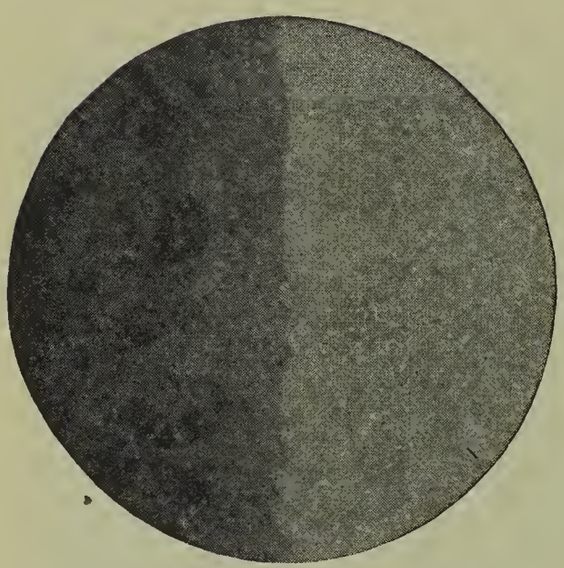

FIG. 9. Cu-Pt. × 1000

FIG. 10. Cu-Ag. × 1000

In welds between some metals diffusion takes place, but in any of the useful combinations the change is too slight to affect the ductility of the weld. The welds as a rule show only a sharp dividing line between the metals but there is often an intermingling of the two at or near the center for a very short distance. Figs. 7 and 8 show a new weld and a three-year-old weld

between aluminum and copper. The micrographs were taken at the irregular point in the weld. Elsewhere the line of division is sharp and rather straight. In addition to the small irregularity of the dividing line some spots of bright material, possibly aluminum-copper alloy, are present at this point, but do not appear at other points in the weld. Both of these welds are so malleable that they are capable of being rolled into thin foil. Such welds, run at a temperature over 100° C. by the passage of heavy direct current, failed to show sufficient diffusion to affect their mechanical properties. The heating current was maintained for weeks and tests were made with both directions of current flow. At higher temperatures (red heat) there was a very rapid diffusion of the metals and in a few minutes the metals were diffused for a distance of 2 or 3 inches.

This type of weld offers a very convenient specimen for the study of the diffusion of different metals at different temperatures and under various other conditions. The micrographs of Figs. 9 and 10 show copper-platinum and copper-silver respectively. Both of these welds show a sharp dividing line at 1,000 diameters and the weld of Fig. 9 is three years old.

It is evident that the electrical resistance of two wires welded together will not be appreciably increased by the small film of high resistance alloy which may be formed in welding. Tests of a wire of 85 alternate pieces of aluminum and copper joined with 84 welds in a total length of 23.5 cm. showed an increase of 0.56 percent in resistance in three years. This test was made to determine whether or not diffusion would occur. The increase is small and may be due to a change in the joints or may be due to error of observation or oxidation of the wire. The sample was recently rolled and its malleability indicated no appreciable change.

Applications.—While the development of this welding method was brought about primarily by the necessity for the successful joining of aluminum wires and the joining of copper leads to such aluminum wires, it is evident that the method is capable of an extremely varied application. Since metals varying as widely in characteristics as platinum and tin may be easily welded, it follows that almost any metals may be joined where the joint is within the limits of the capacity of the welding apparatus.

While the apparatus up to this time has been made only for the welding of wires, 0.072 inch diameter and smaller, the application to larger sizes is merely a question of the design of suitable apparatus. Enough has also been done to show that welding other than the welding of wires may be successfully accomplished; as for example, the welding of points to plates and small pieces to irregular shaped objects or flat surfaces.

While it is not the intention of this paper to outline the possible applications of this method, it may be said that electro-percussive welding has already been used to a considerable extent in the joining of aluminum wires; the welding of copper and aluminum wire; platinum and nickel; platinum and copper; and the welding of various types of thermo-couple wires. It has also been used for the reclamation of short pieces of wires of various kinds, such as aluminum, platinum, spring steel, etc.

The electro-percussive method is suitable for many applications in the jewelry trade, such as the joining of platinum without showing any solder line; the welding of sterling tips to table forks without annealing; the welding of pins to badges, and many other similar applications. The attachment of contact points of platinum, tungsten, silver, etc., for various electrical purposes is also very readily accomplished.

In conclusion, the authors believe that the electro-percussive method of welding opens up a wide field of welding hitherto impossible. It also overlaps, to a certain extent, some of the present methods, but it will probably never supersede the existing methods for the welding of very large sections.

DISCUSSION.

JOS. W. RICHARDS: Has Mr. Chubb used that method with thermo-couples of several sizes?

L. W. CHUBB: Yes, we have used it a great deal with thermo-couples of various types, such as platinum, platinum-rhodium, copper-constantan, etc. We have also reclaimed lots of short ends of platinum and platinum-rhodium wire by welding them

together. The couples have also been made by putting the two metals together, rolling them sideways and making a surface couple, backed with felt.

G. A. ROUSH: Can you give some idea of the cost of an apparatus like this?

L. W. CHUBB: I could not. You could probably tell as well as I how much it would cost to machine these parts and put it up. The condenser itself is very cheap. I do not know anything about the cost end of the business.

G. A. ROUSH: Nothing has been done about putting it on the market?

L. W. CHUBB: Probably steps will be taken to market the apparatus very soon through our company or some other company.

M. G. LLOYD: What is the largest sized wire you have welded so far?

L. W. CHUBB: A No. 13 B & S wire we have tried and have chucks for.

JOS. W. RICHARDS: Have you welded together a metal which has as high a melting point as tungsten?

L. W. CHUBB: We have welded tungsten and we have welded softer metals and tungsten together. Tungsten is so hard you must have a pretty heavy hammer to forge the metal properly. We have not made a machine for tungsten, but in our experiments we have found it would weld.

JOS. W. RICHARDS: Would a malleable metal, like zinc, weld to another metal?

L. W. CHUBB: We have welded zinc, but it crushes a good deal; it is not sufficiently ductile in wire form.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 1, 1914, President F. A. Lidbury in the Chair.

SOME EXPERIMENTS ON THE QUANTITATIVE DETERMINATION OF RADIUM.

By Herman Schlundt.

The accurate determination of radium in radium-bearing ores is generally conducted by the emanation method. With proper care the method is very certain and delicate. It is based upon the fact that the gaseous disintegration product of radium, the emanation, can be completely separated from radioactive materials, and that the equilibrium quantity is proportional to the content of radium. The minute quantities of radium emanation concerned in quantitative determinations of this kind are capable of exact measurement by electrical methods. The emanation is introduced into an air-tight electroscope or an ionization vessel connected with an electrometer, and the saturation current produced, under proper conditions, is proportional to the quantity of radium in the specimen from which the emanation was separated. The measuring instruments are standardized by means of known quantities of emanation from a standard radium solution, or from weighed amounts of uraninite or some other uranium-bearing mineral not of secondary origin, whose uranium content is known. In standardizing electroscopes by the latter method the radium content of the mineral is obtained by multiplying its uranium percentage by the proportionality factor 3.4×10^{-7}.

In the assay of radium ores the complete separation of the emanation from the specimen under examination is of course absolutely essential for accurate work. The collection and subsequent transfer of the gas is largely a matter of careful manipulation. The work of various investigators has shown that heating even to a white heat rarely expels all of the emanation from solids. The emanation, however, is completely liberated

from the ore by dissolution and boiling. For substances soluble in water or acids several forms of apparatus for the quantitative separation of the emanation have been described.[1]

Rocks and minerals that do not dissolve in acids upon heating, or are only partially soluble in them, require different treatment. One of the standard methods, known as the solution method, consists in fusion of a definite quantity of the material with four to five times its weight of mixed carbonates of sodium and potassium. The fused mass is lixiviated with water and washed with dilute sodium carbonate solution. The insoluble residue is dissolved in dilute hydrochloric acid. The alkaline and acid solutions are placed in separate flasks which are corked. On standing for a month the equilibrium quantity of emanation is again produced by the radium in solution. The emanation is then separated by boiling, and transferred to an emanation electroscope, and after three hours, when the activity is a maximum, the saturation current is measured.

Recently Joly[2] and others have introduced fusion methods which appear to be more reliable than the solution method. The emanation is released from the rock by fusing it with a suitable flux—mixed carbonates of sodium and potassium are generally used—in a closed, electrically heated furnace at a temperature of over 1,000° C. The liberated emanation is collected, and after being freed from carbon dioxide, is transferred to a standardized electroscope for measurement.

The results obtained by the fusion method are generally higher than the values by the solution method. In the solution method it is difficult to avoid the formation of slight precipitates in the solutions, and these doubtless cause the separation of some radium, which does not release its emanation completely upon boiling.

An electric furnace of the Joly type would serve very well for the assay of radium ores that are not completely soluble in boiling acids, and it would be particularly well suited for ores of very low grade, as charges of thirty grams can be readily fused in the ordinary size of furnace. The furnace, however, is of special design, a large platinum crucible is required as well as heavy

[1] The forms of apparatus used by Strutt, Boltwood, McCoy, Schlundt and Moore, are illustrated and described by Randall, Trans. Am. Electrochem. Soc., **21**, 463 (1912).

[2] Joly, Phil. Mag. **22**, 134 (1911).

platinum wire for the heating resistance. Various electrical measuring instruments and other electric accessories are desirable parts of the equipment. On the whole, the apparatus of Joly is rather expensive equipment for an assay laboratory. Our experiments were directed toward devising and testing the accuracy of a simple fusion method for the assay of even low-grade radium ores, that can be conducted with inexpensive apparatus.

We have found that charges as large as twelve to fifteen grams can be successfully fused in hard glass test tubes made of combustion tubing of large bore. The method will thus serve for the determination of radium in very low-grade ores. The specimen of carnotite ore used in our experiments had a radium content equivalent to only 0.6 percent uranium. A two-gram sample of the ore when fused with ten to twelve grams of mixed sodium and potassium carbonates furnishes sufficient emanation for an accurate determination, notwithstanding the fact that the continuous loss of emanation under ordinary conditions is over 40 percent. A fusion tube in the form of a test tube 25 centimeters long affords ample space for a 12-gram charge. The open end of the tube is fitted with a two-hole rubber stopper carrying glass tubes, one of which extends down the tube to within a few centimeters of the charge. One of the tubes is closed during a fusion and the other is connected to an absorption tube and a special form of gas burrette of about 200 cc. capacity connected with a leveling reservoir. The apparatus (Fig. 1) used for collecting the gas was of the form used by Moore and the writer, and by Randall in the estimation of radium by the method of boiling. The flask *A* is not used in the fusion method and the clip at *E* is therefore closed. The fusion tube is attached on the side *T*. In most of the experiments the absorption tube contained a few pieces of sodium hydroxide and a sufficiently small volume of water to permit the back flow of gas without drawing liquid into the fusion tube. For smaller charges side neck tubes of smaller bore were used. Such tubes are easily made from combustion tubing in an oxygen-gas flame. In some of the experiments hard glass tubes drawn out to a tip were used. After fusion is completed the emanation in the tube can then be transferred to the electroscope by simply breaking the tip and passing a current of air over or through the melt. In the decomposition

of ores by boiling with concentrated sulfuric acid, the fusion tube was replaced by a round-bottom flask of about 100 cc. capacity. When fluxes evolving oxygen were used the absorption tube was replaced by a hard glass tube containing copper gauze which was heated during fusion.

In conducting a determination the gas burette is first filled with freshly boiled water. The fusion tube containing its charge is attached at *T* and the other outlet of the tube is closed. All the

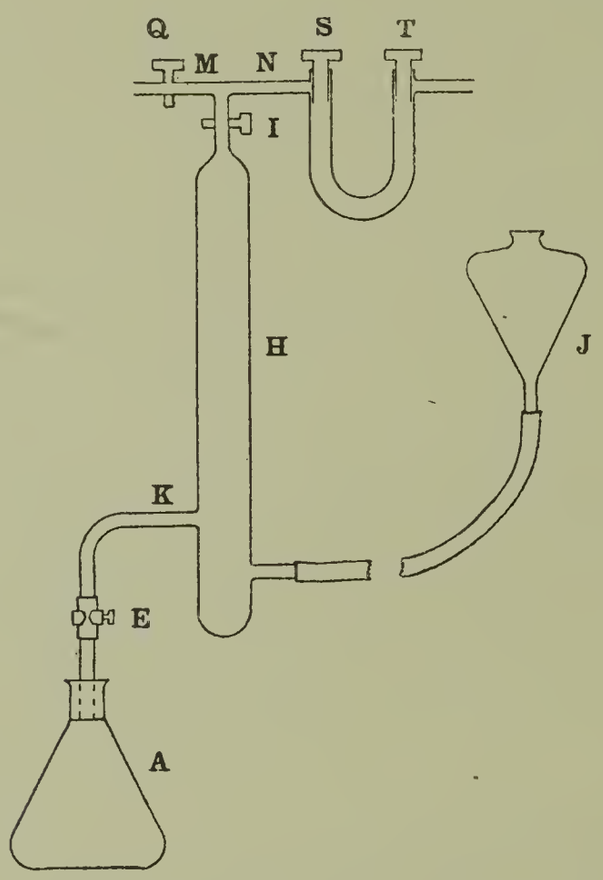

FIG. 1. Schlundt and Moore Apparatus.

glass stop-cocks except *Q* are then opened and the leveling reservoir is lowered to the position shown in the figure. Any air leak in the apparatus is thus readily detected. The fusion tube should be inclined slightly upward from the horizontal so that the melt will flow toward the closed end. Freshly heated fluxes should be used and the entire tube should be warmed first to minimize the danger of reflux water drops cracking the tube. During fusion the water level in the reservoir is adjusted from time to time and is always maintained at a little lower level than the water in the burette. A slight excess external pressure is

thus maintained on the fusion tube during heating, until it gradually collapses at the hotter parts. The heating is done with a large-size Meker burner aided by a hand blast lamp. The heating of the melt is continued for fifteen minutes. When the fusion is made with mixed carbonates there is a small, steady evolution of gas all the time during the heating as the result of the action of the flux on the glass tube. This continuous evolution of gas doubtless serves a very useful purpose in removing the emanation from the melt. In our experiments with a carnotite concentrate consisting largely of calcium sulphate, a little powdered quartz was added to the charge to increase the evolution of gas during fusion.

While the tube is still hot an exhausted electroscope is attached at *Q*, and the gas in the burette is drawn into the electroscope until water reaches the tube *MN*. The stop-cock *I* is then closed and the inlet tube of the fusion tube is opened. The remaining gas in the tube and absorption train are thus swept into the electroscope.

Three hours after the introduction of the gas into the electroscope the ionization current reaches a maximum value which remains constant for nearly half an hour. Readings are made at this time to determine the rate of discharge. By keeping the leaf charged from eight to ten minutes before readings are to be made very uniform rates of discharge are obtained.

Two electroscopes were used in our experiments. One was a gold leaf instrument[3] and the other a Wulf quartz fiber electrometer.[4] In using the Wulf instrument it was found that the ionization chamber can withstand pressures of two-thirds of an atmosphere. The gas was therefore introduced directly into the exhausted chamber and the circulation method recommended by the inventor was not used. Both instruments were standardized by means of known quantities of radium emanation separated from weighed amounts of uraninite containing 53.03 percent of uranium. The emanation was separated in a small flask like *A*, Fig. 1, by dissolving in nitric acid (1 : 1) and boiling. The flask in these calibration experiments was fitted with a two-hole rubber

[3] The instrument is illustrated and described in Randall's paper, Trans. Am. Electrochem. Soc., **21**, 477.

[4] Wulf, Physik. Zeitschrift, **8**, 246, 527; **10**, 251.

stopper, one of which held a small drop funnel.[5] Our specimen of uraninite continuously emitted 2.6 percent of its emanation at room temperatures. We have adopted the plan of keeping on hand a stock of weighed samples to be used for calibrating electroscopes, stored in the small flasks at least a month before boiling off the emanation. The total equilibrium quantity of emanation is thus obtained.

As most radioactive solids continuously emit a portion of their emanation at room temperatures, the quantity of emanation released by fusion or boiling is less than the equilibrium quantity. The emanation loss in the cold was determined by enclosing a definite weight of the powdered ore, ground to pass through an eighty mesh sieve, between layers of cotton in glass stoppered U tubes, and allowing the emanation to accumulate for a definite time—a period of four days or more. Then by drawing air through the tube into an emanation electroscope the fraction continuously emitted can be readily deduced. The equilibrium quantity of emanation that will accumulate in the tube was calculated by the equation:[6]

$$E_t = E\,(1 - e^{-\lambda t}),$$

where E_t represents the quantity found after t hours of storage; E the maximum quantity that will accumulate, e, the base of the natural logarithms, and t, the radioactive constant, 0.0075 $(\text{hr})^{-1}$. To ascertain whether samples taken from stock in closed bottles still retain a part of their emanation, after a few minutes of exposure during weighing, several samples were weighed out and placed in U tubes and then air drawn through them immediately into an electroscope. No increase in the rate of discharge of the leaf was observed.

The fusion method of determining radium described above was tested with four radioactive solids: (1) The specimen of uraninite used in calibrating the electroscopes; (2) A composite of a carnotite concentrate which had been obtained from carnotite

[5] For details of operation see Schlundt and Moore, Jour. Phys. Chem., 9, 320 (1905).

[6] Rutherford, "Radioactive Substances and Their Radiations," (1913) pp. 336, 665. Kolowrat, Le Radium, 6, 195 (1909).

ore on a commercial scale by the Fleck method.[7] The concentrate consists largely of calcium sulphate; (3) A specimen of low-grade carnotite ore from the Paradox Valley, Colorado; (4) A spring deposit (sinter) from Doughty Springs, Colorado. The material is a unique spring deposit in that it consists of about 87 percent barium sulphate. This specimen was kindly sent by Dr. Headden of the State Agricultural College of Colorado, who first described this interesting group of radioactive springs.[8]

The radium content of these specimens is given in the fourth column of Table I; the uranium equivalent appears in the last column; and in the second and third columns are recorded the percentages of the equilibrium quantity of emanation emitted at ordinary temperatures and at red heat. The radium content of the uraninite was deduced on the assumption that radium and uranium exist in equilibrium in the specimen. For the analysis of the sample the writer is indebted to Dr. Randall.[9]

TABLE I.

Results of Analyses.

Substance	Percent of Cold Emanation Loss	Percent of Emanation Loss at Red Heat	Ra per g. x 10^9	U per g. x 10^3
Uraninite	2.6	25.6	180.2	530.3
Carnotite concentrate...	18.1	88.6	24.24	21.3
Carnotite ore	41.8	90.	20.77	6.11
Spring deposit	2.8	30.	16.54	4.57

The results obtained for the radium content of the four substances by different methods of separating the emanation, and by means of different fluxes, are given in Tables II, III, IV and V. The numbers in the first column indicate the number of determinations that were averaged to give the values in the third and fourth columns.

[7] Cf. Bulletin 70, Dept. of the Interior, U. S. Bureau of Mines, "A Preliminary Report on Uranium, Radium, and Vanadium," by Richard B. Moore and Karl L. Kithil, p. 74 (1913).

[8] Headden, Proc. Col. Sci. Soc., 8, 1 (1905).

[9] Randall, *loc. cit.*

Table II.

Uraninite.

Number of Experiments	Method of Separating the Emanation	Grams Radium per g. x 10^9	Grams Uranium per g. x 10^2
9	Solution in nitric acid and boiling....	180.2	53.03
6	Fusion with mixed carbonates.....	179.2	52.7
2	Fusion with potassium acid sulfate...	179.9	52.9
2	Fusion with potassium nitrate	180.9	53.2
2	Fusion with sodium peroxide.....	180.5	53.1
2	Fusion with potassium chlorate.....	179.9	52.9
2	Fusion with potassium dichromate....	177.1	52.1
2	Heating in hard glass tube.....	46.6	13.7
2	Boiling with concentrated sulfuric acid	77.2	22.7
2	Fusion with litharge	101.3	29.8
2	Fusion with lead peroxide	136.0	40.0
2	Fusion with borax	165.6	48.7

Table III.

Carnotite Concentrate.

Number of Experiments	Method of Separating the Emanation	Grams Radium per g. x 10^9	Grams Uranium per g. x 10^2
2	Fusion with carbonates, solution and boiling	19.96	5.87
2	Heating in hard glass tube	21.32	6.27
2	Boiling with nitric acid	23.05	6.78
5	Fusion with potassium acid sulfate....	24.24	7.13
4	Fusion with mixed carbonates	24.07	7.08

Table IV.

Carnotite Ore.

Number of Experiments	Method of Soparating the Emanation	Grams Radium per g. x 10^{10}	Grams Uranium per g. x 10^3
2	Fusion with carbonates, solution and boiling	15.85	4.66
1	Heating in hard glass tube.....	18.65	5.31
2	Fusion with potassium acid sulfate....	18.63	5.48
2	Boiling with nitric acid.....	18.33	5.39
2	Fusion with mixed carbonates	20.77	6.11

Table V.

Spring Deposit.

Number of Experiments	Method of Separating the Emanation	Grams Radium per g. x 10^{10}	Grams Uranium per g. x 10^3
2	Fusion with carbonates, solution and boiling	11.29	3.38
2	Boiling with concentrated sulfuric acid	13.97	4.11
2	Heating in hard glass tube.....	4.93	1.45
2	Fusion with potassium acid sulfate...	11.02	3.24
3	Fusion with mixed carbonates.....	16.54	4.57

The results of the foregoing tables point to the following conclusions:

1. The solution method, that is, fusion with mixed carbonates, followed by solution and boiling, gave results that are only 70 to 80 percent of the values obtained by the fusion method with mixed carbonates.

2. Fusion with mixed carbonates appears to be superior to fusion with potassium acid sulphate, although with the carnotite concentrate a slightly higher value was obtained with the sulphate.

3. Fusion with borax probably gives low values as a result of the retention of some of the emanation in the melt. At the temperatures attained in our experiments the evolution of gas and its flow through the melt is necessary for complete release of the emanation.

4. High emanating power in the cold indicates a large release of emanation by heating. In the case of the specimen of carnotite ore a rough assay may be made by merely heating the ore to bright redness.

5. A complete separation of the emanation from uraninite is obtained by solution in nitric acid and by fusion with some of the ordinary fluxes.

No experiments were conducted with the fusions to ascertain whether the equilibrium quantities of emanation can be again separated by a subsequent fusion. Experiments similar to the ones here presented have been conducted in this laboratory during the past year by Dr. W. G. Brown and Mr. O. E. Sheppard in connection with an extensive series of experiments with a Joly furnace on the radium content of rocks.

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DISCUSSION.

S. C. Lind (*Communicated*): As Dr. Schlundt has pointed out, the accurate determination of radium by the emanation method resolves itself into the problem of quantitatively removing

emanation from the radioactive substance being analyzed. Consequently the study of a variety of methods for different radioactive substances, as reported in the present paper, forms a valuable contribution to the subject. Incidental to the determination of the radium: uranium ratio in carnotites recently carried out by Mr. C. F. Whittemore and myself,[10] the quantitative de-emanation of carnotite was studied in a large number of samples of ore by four different methods and it may be of some interest to compare our results with those of Dr. Schlundt.

As regards the method of fusion with $Na_2CO_3.K_2CO_3$ mixture and subsequent dissolution and decomposition of the residues in dilute acid, we agree fully with Dr. Schundt in obtaining results 10 to 20 percent low.

The method of dissolving directly in boiling 1-1 HNO_3 was found to give correct results, as indicated by the Ra/U ratio, and also by agreement with a direct ignition method, employing Jena glass and a Meker burner. The result with HNO_3 is in general accord with Dr. Schlundt's, although in the case of carnotite he obtains a somewhat lower value than by fusion with carbonate. The agreement obtained by us in more than twenty cases between the solution and ignition methods in the case of carnotite and supported by the normality of the Ra/U ratios has led us to place the highest confidence in these two methods for this ore, and it is to be noted that Dr. Schlundt's results by these methods also agree with each other for carnotite ore, though somewhat lower than he obtained by the fusion method.

With respect to the carbonate fusion method, we have failed in all carnotites to obtain anything approaching complete de-emanation, which result we have been inclined to attribute to the influence of silicates on the viscosity of the melt, because the same procedure has been successfully employed by us for pitchblende and crude sulphates containing little or no silica. The marked contrast with Dr. Schlundt's results for this method would indicate that it is primarily a question of temperature or gas evolution although the latter had been abundant in our experiments also.

Although our experiments were mainly confined to carnotites, some results obtained with pitchblende and crude sulphates have

[10] Lind, S. C., and Whittemore, C. F., Jour. Amer. Chem. Soc., Oct. 1914.

led us to believe that a method may be highly specific in its applicability to a given radioactive substance. For example, the ignition method, successful with carnotites, is a complete failure under the same conditions for crude sulphates; and the fusion method, as already stated, succeeded with pitchblende but failed with carnotites. These considerations seem to render the results and methods of an investigation such as the present one of Dr. Schlundt's all the more valuable, in offering a variety of methods of attacking the different radioactive substances that may present themselves for analysis.

It seems highly desirable to extend the investigation of the method to that of initial de-emanation followed by accumulation and a second de-emanation, which is the only way in which the radium determination may be made independent of the "emanating power" of the radioactive substance, and at the same time be confined to a reasonable length of time. The precautions necessary in determining radium by the use of "emanating power," especially in the case of substances in which the latter has a high value, as in carnotite, has been pointed out in the paper already referred to.[11]

WARREN F. BLEECKER: The measurement of radium is essentially an indirect method quite difficult to make and, except by those expert in the matter, is little understood; and what we are after is a method which is easily worked and which gives reasonably accurate results.

The original method probably was not applicable to all classes of ore, without previous examination; that is to say, each class of ore under examination would first have to be run by a solution method, and the blank determined. For example, Dr. Schlundt makes the statement in his paper that the material must be entirely in solution. That is because a portion of the emanations are held back in solid matter, especially in gelatinous or colloidal material which may remain undissolved. The various elements which are found in radium-bearing rocks have the power of holding emanation in different degrees, and for that reason it would probably make a difference in the quantity of emanation which could be recovered by a fusion method.

[11] Lind and Whittemore, *loc. cit.*

Another thing I wish to call attention to is the fact that a fixed ratio between uranium and radium only holds in old rocks. It does not hold, so far as my experience goes, in rocks which have been in any wise treated, either mechanically or chemically. If you treat a rock chemically, you will dissolve more or less uranium and more or less radium. Mechanically treating it will have the same effect, so one cannot calculate with a slide rule from the uranium content the radium content, or *vice versa*. This matter is of considerable importance to us, because our people are producing considerable quantities of radium, something like fifteen grams a year at the present time. I am sorry I could not bring any radium along with me, but I have here a specimen of uranium carbide produced in the electric furnace. I will show you this, as being a matter of interest in connection with radium. There is much evidence that this material is, if not the parent of radium, the grand-parent.

This substance is of interest and its properties at the present time are little understood.

At one time after making some ferro uranium, I put a sample of it into a box and upon looking into the box through a curtain, saw myriads of stars shooting from the metal. This continued for 48 hours. I have never been able to duplicate this phenomenon, but it may be of great importance, and should be studied. This uranium carbide, as you know, is pyrophoric, due to oxidation. It is, unfortunately, very fragile.

W. R. Mott: To what extent would Dr. Schlundt apply this method to monazite sands which contain mesothorium which is more radioactive than radium?

I also ask the last speaker whether he has run across any trouble from uranium poisoning, because Moissan in his Dictionary on Chemistry, says uranium is one of the most poisonous of the elements (Moissan, Traité de Chimie Minérale, Vol. IV, page 857). Woroschilsky used double tartrate of uranium and sodium, and caused death with a dose of 0.5 to 2 mg. per kilogram with the characteristic appearance of sugar in the urine.

Warren F. Bleecker: A short while ago my assistant and myself got quite a dose of uranium chloride, but we felt no bad effects from it. I have been handling uranium solutions for

years, and have felt no bad effects from them, nor have I ever seen a case of either uranium or vanadium poisoning.

J. W. RICHARDS: I understand that the workmen who have been sampling the ores from the carnotite bearing rocks have become poisoned by apparently absorbing the dust from these ores. The form of poisoning was quite serious. None of the doctors who were called in could diagnose the disease.

WARREN F. BLEECKER: I should like to combat most seriously any notion there might be as to the poisonous effect of uranium. We handle a good deal of it. We have workmen who are covered with it, sometimes from morning to night, and we have never had a single case of poisoning. And if there had been such cases, we would have heard about it, because men are sometimes looking for trouble, but we have not had one case of poisoning. There is trouble in handling dust of any kind, dust in a coal mine or cement works, or anywhere else. Dust is dust, and it is bad. Outside of the fact that these things are dusty, there has come to my attention no specific case of poisoning.

I saw an article in the Journal of the American Medical Association a year or so ago, in which some doctor described the poisonous effects of vanadium sesquioxide in commercial plants. Evidently he did not know his chemistry very well, because this oxide is one of the most difficult things to prepare.

W. R. WHITNEY: I move that the President appoint a committee consisting of Mr. Bleecker and Mr. Mott to get at the facts of this subject. Our Society cannot be satisfied with anything but the authentic determination of a question which brings up points of this character. It is a serious matter for any company manufacturing a product, to have someone stand in the meeting here and say that this substance is said to be poisonous.

J. W. RICHARDS: I will second the motion, and amend it by adding the name of Dr. A. R. Ledoux, who is a member of our Society, as one of the committee to investigate the poisonous effects ascribed to uranium and vanadium. I second the motion with that amendment.

The amendment being accepted by Dr. Whitney, the President put the motion to vote, and it was carried. (Dr. Ledoux having

declined to serve on this committee, Chas. Baskerville was appointed in his place at a subsequent meeting of the Board of Directors.)

W. G. Brown (*Communicated*): Dr. Schlundt refers to some experiments conducted by the writer and Mr. O. E. Sheppard.

Since these experiments dealt with the same problem as Dr. Schlundt's, the determination of radium, a brief resumé of the work may be interesting.

Mr. Sheppard undertook the examination of a series of rock specimens obtained in drilling an artesian well upon the campus of the University of Missouri for their radium content. The specimens were taken every five feet to a depth of about 770 feet.

Before commencing the systematic examination of the specimens he carried out an extensive series of preliminary experiments to determine the most reliable and rapid method for the estimation of radium and the calibration of the electroscope, using various forms of apparatus, materials, and processes to obtain the radium emanation.

First he used a modification of Joly's apparatus for fusions and with this furnace he tried a mixture of potassium and sodium carbonates, a mixture of potassium and sodium hydrogen carbonates, and borax. He used the original absorption apparatus of Joly for the carbon dioxide, afterwards an absorption train devised by himself. He caught the emanation in a rubber bag, following Joly, and then transferred it to the electroscope; afterwards he discarded the rubber bag and collected the emanation directly in the electroscope, which had been exhausted. He used for heating the furnace platinum wire and afterwards nichrome wire. After having made a hundred and twenty fusions by Joly's method and modifications he rejected it and tried—

Second, a solution method, dissolving the material in dilute nitric acid and transferring the emanation, without storing, immediately into the exhausted electroscope by a stream of carbon dioxide and air through an absorption train of potassium hydroxide solution, solid potassium hydroxide and sodium peroxide.

Thirdly, he fused the material with carbonates in a hard glass

tube as described in Dr. Schlundt's paper, using the same absorption train as in his experiments by solution in nitric or other acid.

With regard to the fusion method in hard glass tubes, Mr. Sheppard describes his method of procedure in his thesis on "A comparison of Methods of Determining the Amount of Radium in Rocks and Minerals," submitted to the Graduate Faculty of the University of Missouri in June, 1914, as follows: "For a calibration fusion 10 grams of mixed carbonates were mixed with 5 grams of powdered silica and a small amount of uraninite. After the fusion air was used to sweep the emanation into the exhausted electroscope."

Comparing the methods, Mr. Sheppard says that the chief objection to Joly's method is the time required, that he was hardly able to finish a fusion in less than three hours—tho Joly seems to have taken only about an hour for the fusion when using the mixed carbonates. By using borax in place of the carbonates the time of fusion is lessened and the "borax is more efficient than the mixed carbonates in getting off the emanation."

The solution method in nitric or other acid is "a very rapid method in getting off the emanation." In most cases "15 minutes to a half hour was sufficient to get the emanation from the dry finely ground substance into the electroscope."

The chief objection to it is that nitric acid will not dissolve all substances, although "all the emanation can be driven off from some substances, *c. g.*, carnotite, by boiling with nitric acid even though the solution is not complete."

The fusion in hard glass tubes requires care and practice in the fusion and in the subsequent transfers of the emanation to the electroscope. It "can only be used for small quantities—less than 10 grams—of a substance." "Also from my limited work it seems to offer no advantage over the nitric acid method and in fact is not nearly so efficient in getting the emanation from uraninite" as that method.

For the statements and conclusions concerning the different methods Mr. Sheppard gives the results of 233 determinations in his thesis.

As some of these results are with portions of the same specimens used by Dr. Schlundt, which specimens Mr. Sheppard obtained from Dr. Schlundt, I quote them for comparison:

Doughty Springs Deposit.

(Dr. Schlundt's Table V.)

	Grams Radium per Grams Sample
Joly's Method	1282.4×10^{-12}
" "	$1110. \times 10^{-12}$
" "	$1091. \times 10^{-12}$
Nitric Acid[1]	162×10^{-12}
With Sulphuric Acid, 2 determinations average.	1345×10^{-12}
Fusion in glass tube, 3 determinations average.	1416×10^{-12}

(Colorado) Carnotite Concentrate.

(Dr. Schlundt's Table III.)

Nitric Acid Method, aver. 29 determinations	18230×10^{-12}
Emanation given off in the cold	4054×10^{-12}
Total emanation	22284×10^{-12}
Fusion in glass tube[2]	18090×10^{-12}
" " " "[3]	15212×10^{-12}
" " " "[3]	14495×10^{-12}
Concentrate stored a month	18647×10^{-12}
" " " "	19041×10^{-12}

[1] Not decomposed by nitric acid but by strong sulphuric acid.
[2] Tube containing air before fusion.
[3] Tube exhausted before fusion.

Among Mr. Sheppard's conclusions are the following bearing on the methods of determining radium.

6. "Boiling off with nitric or with some other acid as necessity demands seems a very promising way to separate the emanation from rock and mineral samples. This method seems especially good for carnotite. Solution in an acid seems to liberate all the emanation so that it can be driven off by boiling."

7. "Fusion with carbonates in a hard glass tube as suggested by Dr. Schlundt seems a possible method for carnotite. It will not do for minerals or rocks with very low radium content where a large sample must be used."

H. Schlundt (*Communicated*): The fact that Messrs. Lind and Whittemore have found the normal value for the ratio between uranium and radium in twenty specimens of carnotite, and also agreement in the value by the ignition and solution methods, speaks very strongly for the reliability of these methods in the determination of radium in carnotites. Their forthcoming paper on this subject will thus dispel the uncertainty that has existed regarding the U/Ra ratio in these ores. The lack of agreement between the results of Messrs. Lind and Whittemore

and our experiments, by direct fusion with mixed carbonates, suggests the desirability of conducting further experiments by this method in very hard glass tubes with the hope of obtaining agreeing results by the three methods.

Mr. Sheppard's determinations by solution in sulphuric and nitric acids are in fair agreement with the results of my experiments by the use of these solvents, but his lower values by fusion with mixed carbonates stand in direct contrast to the higher values of my experiments. Mr. Sheppard also obtained low values by de-emanating uraninite by fusion with carbonates, while my experiments by solution in nitric acid and fusion with several fluxes gave closely agreeing values, and in this respect agree with the results of Messrs. Lind and Whittemore.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 2, 1914, President F. A. Lidbury in the Chair.

SILICIDIZED CARBON—SILFRAX.

By F. J. Tone.

When solid carbon is acted upon by vapors of silica or silicon there is formed a silicon carbide product of the same shape as the original carbon but differing from carborundum in essential characteristics. To distinguish this form of silicidized carbon commercially, it has been given the name "silfrax." The properties and characteristics of the product vary with the temperature of formation. If the carbon article is heated in the amorphous zone of a carborundum furnace the temperature range of this zone being 1,550 to 1,820°, the product is amorphous in appearance, showing no crystals except under high magnification. It has a yellow color and a lusterless fracture. If the carbon is heated in the crystalline zone of the furnace, the temperature of this zone ranging from 1,820 to 2,220°, the material is of great density and plainly crystalline. Upon fracture it shows a steel black color and a metallic luster.

The depth of the silicidizing action is mainly a function of the time of reaction and the concentration of silicon vapor present in the furnace atmosphere. To obtain the best conversion a large excess of silicon vapor must be present and the mixture should contain silica largely in excess of the theoretical carborundum mixture. Under regular conditions with articles of pure carbon the maximum penetration is about ½ inch. Articles composed of a mixture of carborundum and carbon can be readily converted to a greater depth.

Chemical analysis of the two varieties made in the amorphous and crystalline zones show that both are silicon carbide and give no basis for regarding the products as new compounds of silicon and carbon. Microscopical examinations show in all cases the presence of the typical hexagonal crystals of silicon carbide.

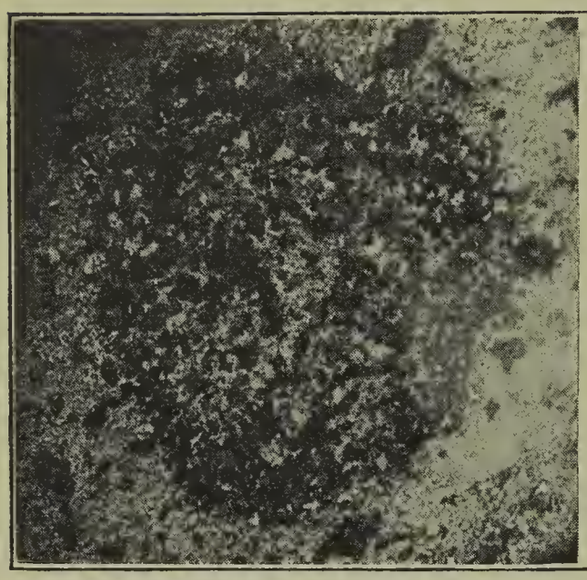


FIG. 1—A. 15 Diam.

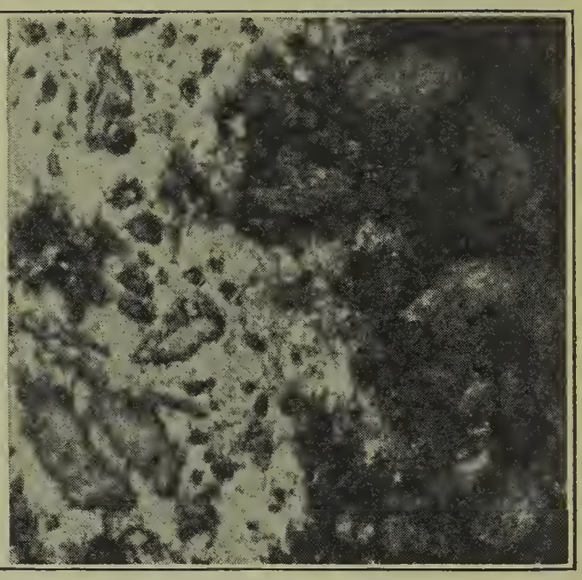


FIG. 2—A. 125 Diam.

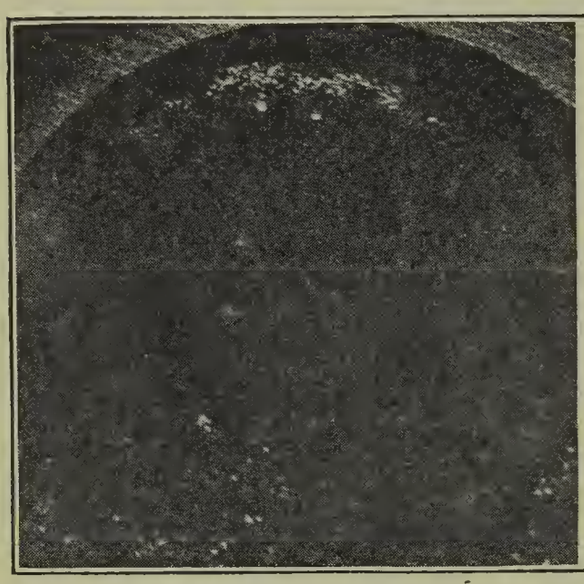


FIG. 3—B. 10 Diam.

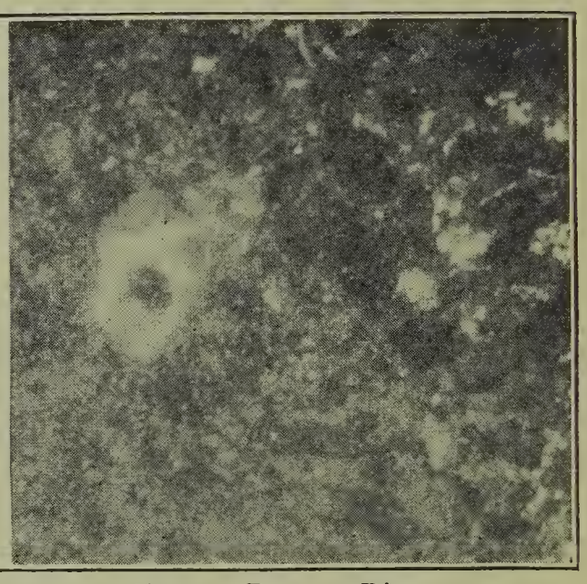


FIG. 4—B. 125 Diam.

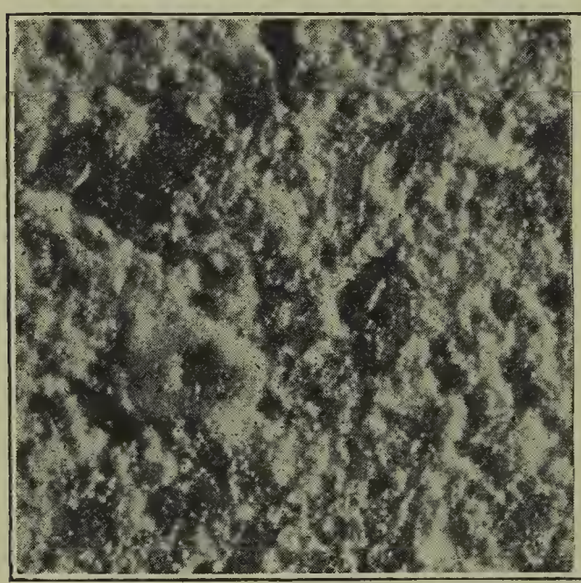


FIG. 5—B. 125 Diam.

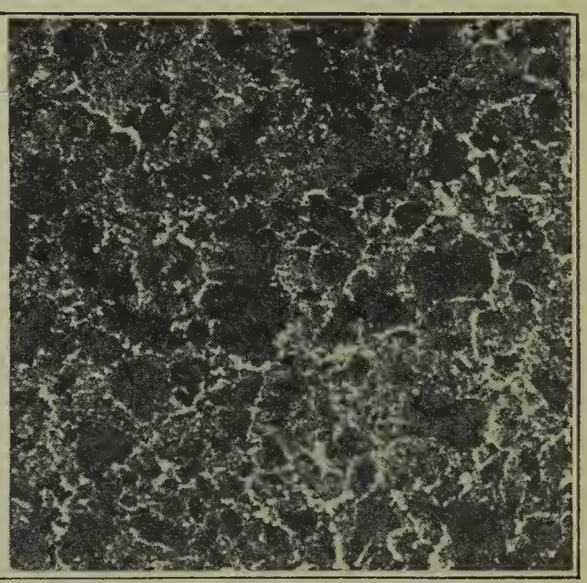


FIG. 6—C. 25 Diam.

Analyses of Silicidized Carbon.

	I	II	III	IV	V
SiC	93.33	91.03	93.39	96.86	94.19
SiO_2	1.14	4.63	0.75	0.94	3.10
C free	1.71	1.16	2.42	0.00	0.48
Fe and Al	2.39	2.44	1.27	1.42	1.67

I. Silfrax silicidized in amorphous zone of carborundum furnace.
II. Silfrax silicidized on edge of crystalline zone of carborundum furnace.
III. Do.
IV. Silundum.
V. Do.

Analyses of the SiC residue show it in all cases to consist of silicon and carbon in exact atomic proportions for SiC, and there can be no doubt that silicidized carbon by whatever name it may be called is silicon carbide.

The following micrographs show sections of carbon rods heated in both the amorphous and crystalline zones of the silicidizing furnace.

Fig. 1. Specimen A; 15 Diam. Ground and polished section of 3/16″ carbon rod heated in the amorphous zone of a carborundum furnace. Color greenish white with small black core and wholly amorphous in appearance. With direct illumination there is much reflected light.

Fig. 2. Specimen A; 125 Diam. Shows structure at junction of inner and outer zones. The outer zone is the lighter and consists of a white ground with irregular grains imbedded in it. The dark spots are soft spots worn hollow in polishing. The grains in the inner zone are larger and crystalline in character.

Fig. 3. Specimen B; 10 Diam. Direct Illumination. Shows 3/16″ carbon rod heated in the amorphous zone but slightly higher than specimen A. Thin greenish exterior and dark core. Amorphous in appearance and possessing no metallic structure. Specimen contains some hard crystals in a soft ground mainly near the outside of the rod, and these being polished by grinding appear as bright spots in this and the following photo.

Fig. 4. Specimen B; direct light, and Fig. 5, oblique light; 125 Diam. Shows outer zone of specimen, the bright spots being crystalline. The surface is too rough to permit of high magnification. Fig. 4 shows a well defined hexagonal crystal.

Fig. 6. Specimen C; 25 Diam., and Fig. 7, 50 Diam., show a

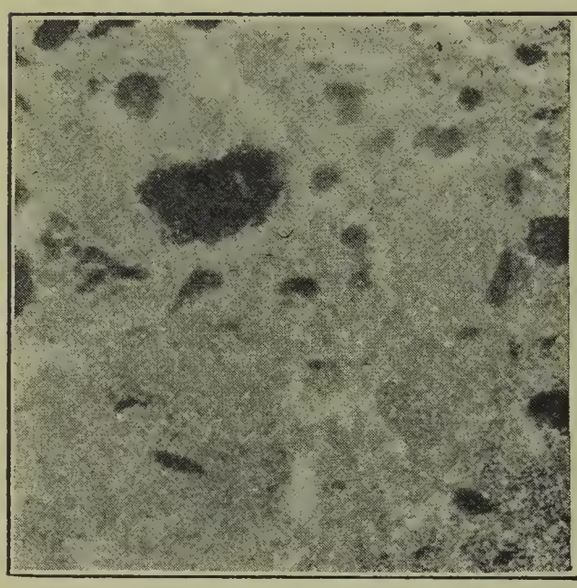


FIG. 7—C. 50 Diam.

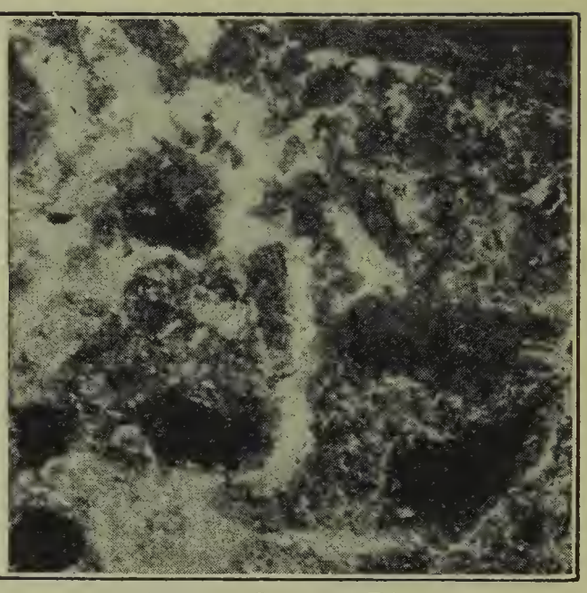


FIG. 8—C. 125 Diam.

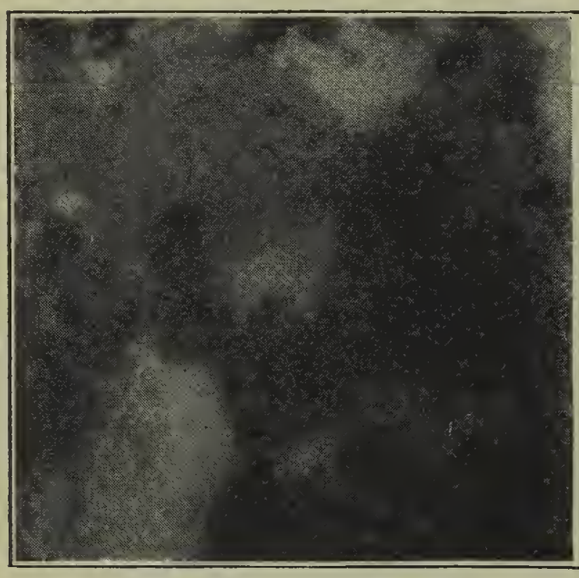


FIG. 9—C. 600 Diam.

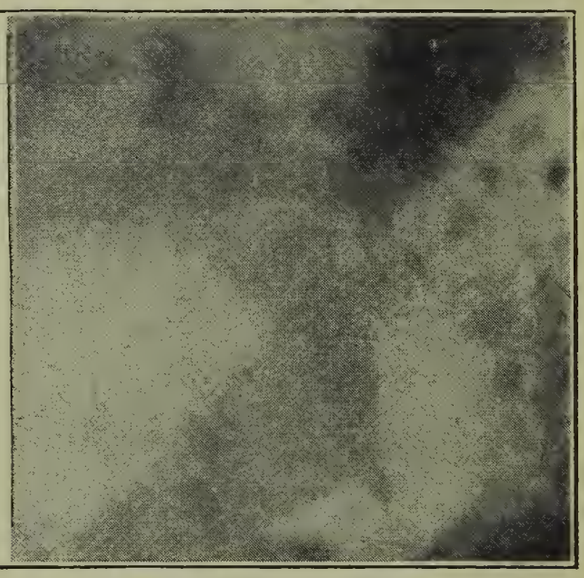


FIG. 10—C. 600 Diam.

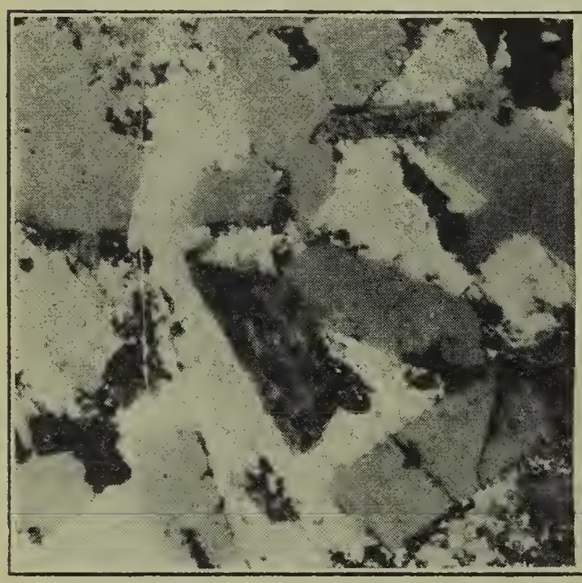


FIG. 11—D. 125 Diam.

FIG. 12—D. 125 Diam.

3/16-inch carbon rod heated in crystalline zone. Color black. Metallic fracture. Photos show black holes, a white network or matrix and the rest of the field gray mottled.

Fig. 8. Specimen C; 125 Diam.

Figs. 9 and 10. Specimen C; 600 Diam. Gray mottled portions are plainly crystalline.

Figs. 11 and 12. Specimen D; 125 Diam. Carbon rod heated in crystalline zone. Color black. Fracture metallic. Rod was polished on emery wheel and finished with rouge. No etching was needed to develop the structure. The photo shows: (a) crystals of light gray color and smooth surface; (b) darker crystals with mottled surface suggesting pits or inclusions; (c) white matrix; (d) black holes.

Fig. 13. Specimen D; 600 Diam. Direct light. Shows the smooth and rough crystals and white matrix.

When carbon articles are subjected to the action of silicon vapors and molten silicon for a sufficient period the carbon is silicidized in the same manner as articles placed in a carborundum furnace, but the resultant product is carborundum containing varying proportions of pure silicon. Carborundum and silicon appear to be mutually soluble in all proportions and the physical and electrical properties of the carborundum are altered by the presence of silicon. A carbon block subject to this action in a silicon furnace furnishes a very suitable subject for investigation. Starting from the interior which is unconverted carbon the following zones are found:

(1) A greenish yellow product (*E*) not of great toughness and readily crushed down to the constituent grains, which are of great hardness. It is a poor conductor of electricity. On treatment with HF and HNO_3 a bright yellow residue is left, which is perfectly crystalline and contains no amorphous material. Even the smallest particles under high magnification are crystalline and practically colorless.

(2) A uniform dense gray product (*F*), having a non-crystalline appearance, very hard tough and difficult to fracture. Treatment with HF and HNO_3 leaves a yellow crystalline material identical in appearance and properties with product *E*.

(3) A coarsely crystalline product (*G*), silver gray in color, very hard but more readily fractured than product *E*. The resi-

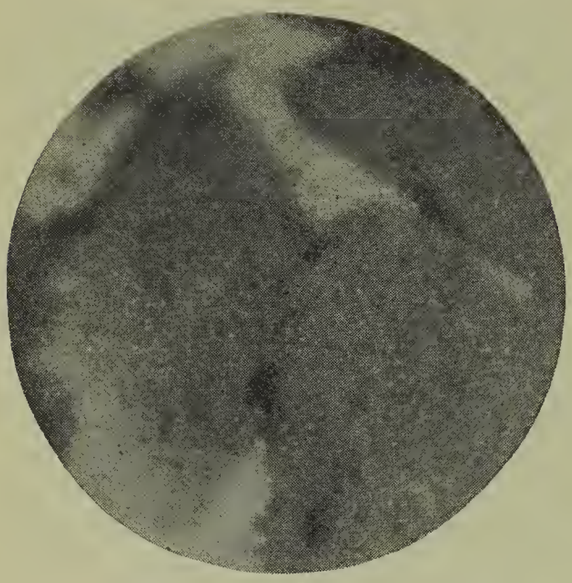


FIG. 13—D. 600 Diam.

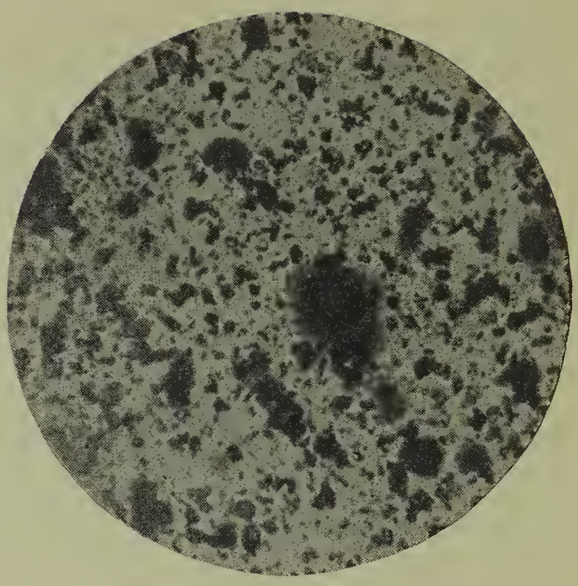


FIG. 14—E. 50 Diam.

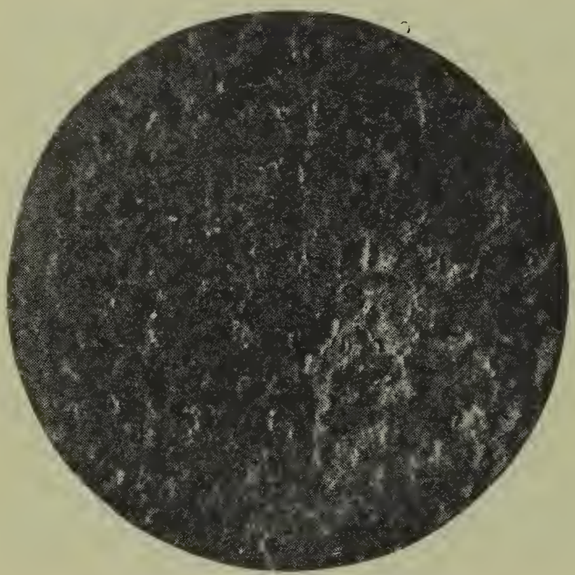


FIG. 15—F. 25 Diam.

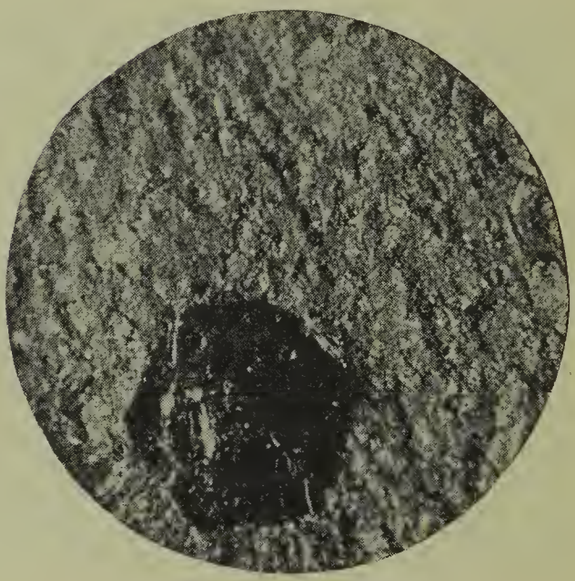


FIG. 16—F. 25 Diam.

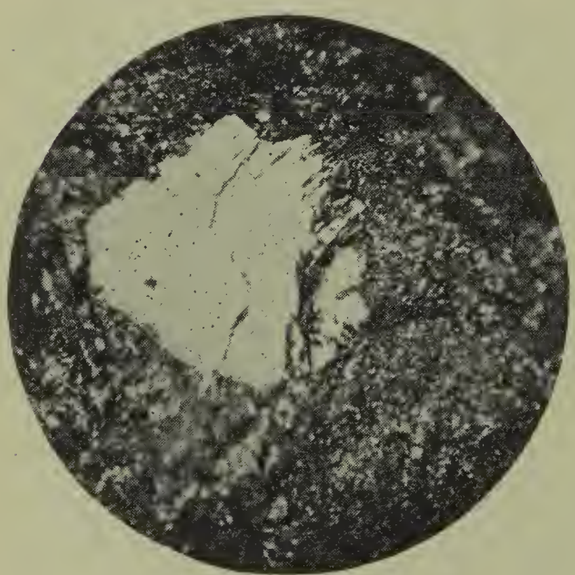


FIG. 17—F. 25 Diam.

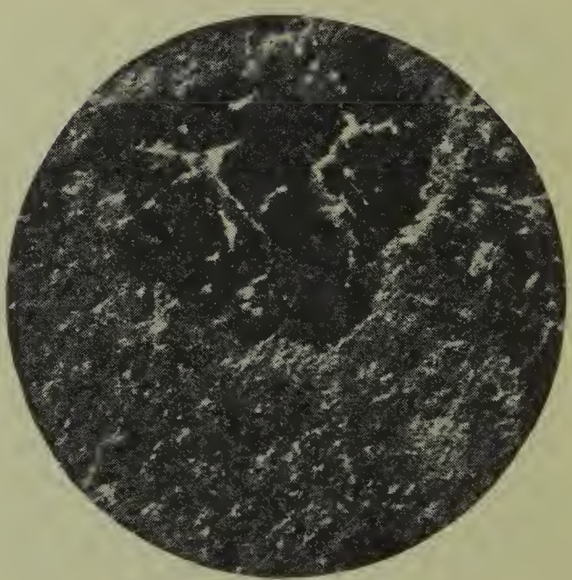


FIG. 18—F. 25 Diam.

due from treatment with HF and HNO_3 is mostly yellow but contains some dark grains and is entirely crystalline.

Chemical Analysis of Products.

	E	F	G
Free Si	7.40	21.27	10.45
SiC residue	91.10	76.10	87.72
Fe	.25	1.40	.93
Al	.45	.57	.36
CaO	.80	....	....
	100.00	99.34	99.46
Residue from HF and HNO_3:			
Si	69.40	....	69.32
C	29.33	....	29.29
Fe and Al	....	....	.10
	98.73	....	98.71

	Apparent Density	S. G. of Residue	Resistivity Ohms cu. cm.
Product E	2.91	3.16	174.0
" F	2.88	....	1.8
" G	3.10	3.16	9.3

The following micrographs were made from products *E* and *F*.

Fig. 14; 50 Diam. Green residue from product *E*, after treatment with HF and HNO_3. Every grain that can be resolved by magnifications of 500 diam. is truly crystalline. The grains are of various sizes and seem to correspond with the original sizes of the carbon grains. Colorless by transmitted light and pale green by reflected light.

Fig. 15; 50 Diam. Polished section of product *F* showing fine grained mottled appearance.

Figs. 16 and 17; 25 Diam. Fracture. Fig. 18; 25 Diam. Polished. Product *F*. Crystals are approximately ½ mm. across and badly fractured.

In discussing the difference between silfrax and ordinary carborundum it is necessary to consider the reactions of the carborundum furnace. The reaction of silica and carbon is one which under varying conditions may be made to give a variety of products. Silicon carbide, silicon, silicon monoxide, siloxicon, graphite and modifications or mixtures of these may all be produced by variations in the mixture proportions of silica and carbon, temperature, pressure, rate of energy expended and other

factors. In the carborundum furnace, carborundum is initially formed by the reaction of silica vapors on carbon beginning at a temperature of 1,550°. The first carborundum so formed is always associated with silica and may be regarded as a definite compound, as siloxicon, or as a mixture or solid solution of silica and silicon carbide. Whatever may be its composition when treated with HF and HNO_3 it is freed from silica and silicon, the residue being silicon carbide usually in the amorphous state.

At a higher range of temperature complete conversion occurs in accordance with the equation:

$$SiO_2 + 3C = SiC + 2CO. \qquad (1)$$

On this equation is generally based the chemistry of the carborundum furnace, but as a matter of fact another reaction plays the most important part in the formation of carborundum and is here offered as a theory to explain fully all the phenomena of the furnace. Equation (1) explains only the conversion of carbon particles into carborundum *in situ,* the crystals or aggregates so formed being no larger than the original carbon particles. It is impossible to explain the growth of large crystals or crystals appreciably larger than the original carbon particles, unless it be granted that both the carbon and silicon when combining to produce these crystals are in the form of vapor. The largest and most perfect crystals are found in cavities or spaces where carbon and silicon in some form of gas or vapor have commingled and combined to form the solid crystals. Such form of carbon, however, cannot be vaporized carbon because it is well known that, at the temperature and vapor pressure of the carborundum furnace, carbon has no appreciable vapor tension. This is further proven by the fact that the effect of high temperature alone is to decompose carborundum, the silicon being vaporized and the carbon being left in a skeleton of the exact form of the original carborundum crystal.

If the vapor tension of carbon was appreciable these graphite pseudomorphs would not retain the perfect shapes of the carborundum crystals so generally observed. The theory is untenable therefore that carbon vapor and silicon vapor unite to form the large crystal growths.

To explain fully all the phenomena of the furnace the following reaction is necessary, the reaction being reversible:

$$2CO + 3Si \leftrightharpoons 2SiC + SiO_2 \qquad (2)$$

Carbon monoxide and vapors of silicon and silica are known to be present throughout the furnace atmosphere, and equilibrium relationships and the direction of the reaction in equation (2) depend on the temperature, pressure and the concentration of the reacting substances. The direct reaction is the low temperature reaction and is exothermic. It occurs throughout the crystalline zone, and on it is based the building up of the large crystals from the small crystals formed by equation (1). The reverse reaction occurs in the hottest part of the furnace and next to the core, and explains the phenomenon of the local disappearance of carborundum. This is observed in the rounded formation of the crystals next to the core, which have lost their sharp outline on account of being washed by the silica vapors. It is further observed in the smooth channels and blow holes cut through masses of crystals by the blowing out of the furnace gases. That carborundum can be decomposed or oxidized by silica is well known and is one of the methods of producing silicon.

Reaction (2) further explains the phenomenon of the recrystallization of carborundum first discovered by FitzGerald. Recrystallization can only occur in an atmosphere of CO, SiO_2 and Si. It is impossible to recrystallize carborundum in a pure atmosphere of CO by the action of heat alone.

Applying these considerations to silfrax it will be seen that it differs fundamentally from ordinary carborundum in its mode of production. It is produced mainly in accordance with equation (1), that is from the action of silicon vapors on carbon *in situ*, and not from gaseous carbon compounds. The crystals composing silfrax therefore are very small and closely aggregated. The development of large carborundum crystals requires free space for the circulation of gases. The largest and most perfect crystals in the carborundum furnace are produced in cavities resulting from the settling of the charge and these cavities are located at a point on the temperature gradient representing approximately the equilibrium temperature for equation (2). In producing silfrax the density of the carbon article is such that

when the initial coating is formed the pores are almost completely filled with SiC, making the further circulation of gases very slow and difficult. Reaction (2) which is vapor reaction cannot occur to any extent and the crystals remain small and closely compacted. Silfrax is a crystalline aggregate while ordinary carborundum is a crystal or crystal aggregate. To this is due the superior toughness and mechanical strength of silfrax.

The commercial applications of silfrax are as refractory materials, electrical heating elements and chemical ware. It has all the chemical and heat resisting properties of carborundum and in addition great density and toughness. Heretofore the articles produced have been of small size. Pyrometer tubes are now made 24 inches in length, and by special furnace construction it will be possible to make much larger sized articles for special refractory vessels and chemical apparatus. It has not yet been determined whether the material can be produced of sufficient density to be used as acid evaporating vessels, for which its very low co-efficient of expansion and high thermal conductivity specially adapt it.

DISCUSSION.

CHARLES B. THWING: I am interested in the matter from the pyrometer side. There are two particulars in which the tubes should be superior to porcelain tubes; they are more refractory and doubtless are better conductors of heat. The one particular of importance is, how impervious to gas the tubes are as compared with porcelain and how long they retain this imperviousness under use at 1200° or 1500°.

JOS. W. RICHARDS: Do these tubes give off silicon vapor which would destroy the couple?

CHARLES B. THWING: That might, possibly, be the case.

F. A. J. FITZGERALD: When I read over the advance copy of this paper a few days ago I found some points that might be criticised and saw that some simple experiments might throw light on these. Moreover, the method of making the experiments seemed to be a suitable demonstration for the Experimental Symposium next Saturday.

Tone offers a theory of the carborundum furnace phenomena which seems to be based on doubtful data. Thus, he says "it is well known that, at the temperature and vapor pressure of the carborundum furnace, carbon has no appreciable vapor tension." I do not think this has been proved or that it is generally admitted. On the contrary, there is reason to believe that not only is the vapor tension appreciable, but we may calculate the relative vapor tensions of the different modifications of carbon.

Forssell has pointed out[1] that the relative properties of the different modifications of carbon, *viz.*: diamond, graphite and amorphous carbon, have been studied very thoroughly by means of the reaction:

$$C + CO_2 \leftrightarrows 2CO$$

Equilibrium changes with rising temperature until at 1000° C. the reaction in the direction of the right member of the equation is nearly complete. But the equilibrium is different according to the modification of the carbon in contact with the gaseous mixture. This made it possible to compute the *relative* vapor pressures of the different modifications of carbon and Forssell finds that at 500° C. the vapor pressure of amorphous carbon is 3.7 times that of graphite, while at 640° C. it is 5.4 times as great. It appears then that the vapor pressures of carbon and graphite are regarded as sufficiently appreciable to be differentiated at comparatively low temperatures.

If a carbon rod is embedded in quartz sand or a mixture of quartz sand and carbon and is heated strongly with an electric current we might expect the quartz to fuse and then reacting with the carbon form a gaseous envelope about it. The silicon gas should act directly on the carbon and silicidize it and should also react with the silica vapor building silicon carbide crystals according to Tone's equation (2).

A graphite rod 6 mm. (0.25 inch) in diameter and 200 mm. (8 inches) long, was embedded in a mass of quartz sand and heated by a current of 200 amperes for 30 minutes. The results found correspond with those indicated by the theory, *i. e.*, the rod was superficially silicidized and in the space between the rod and the fused silica tube some fine crystals of carborundum were found.

[1] U. S. Patent No. 983,887.

Now still assuming the same theory, if the carbon rod is embedded in a mass of pulverized silicon we would expect the latter to fuse around the rod and silicidize it superficially. When this experiment was tried it was found that while there was a superficial silicidizing of the rod the surrounding silicon was largely converted into silicon carbide to a distance of nearly 20 mm. (0.8 inch) from the rod.

Here the theory seems to be inapplicable and we would be inclined to believe that carbon vapor from the rod had combined with the surrounding silicon. However, if we still wanted to support the theory we might argue that in the somewhat porous mass of pulverized silicon there was enough air entrapped to oxidize the silicon to silica required by equation (2).

This suggested the use of a graphitized carbon tube 17 mm. (0.7 inch) outside, 13 mm. (0.5 inch) inside diameter and 150 mm. (6 inches) long. The tube was filled with finely pulverized silicon and the ends closed with graphite plugs. The silicon was moistened with a little kerosene oil so that when the tube was heated this would vaporize and wash out the entrapped air. The tube was then embedded in pulverized charcoal to protect it from oxidation and heated with a current of 260 amperes. It was found that the tube was partly silicidized on its inner surface and the silicon largely converted into silicon carbide.

In further support of the theory Tone says: "it is impossible to recrystallize carborundum in a pure atmosphere of CO by the action of heat alone." If this means that by heating carborundum in an atmosphere of CO to the temperature at which it forms from the usual mixture of silica and carbon, then the following experiment should not show recrystallization:

A graphite rod 6 mm. (0.25 inch) in diameter and 150 mm. long was embedded in carborundum powder containing a slight excess of free carbon and heated with a current of 250 amperes. It was found that the carborundum was recrystallized, the part next the rod being converted into graphite.

If we still want to support the theory we might argue that the temperature being so high next the carbon rod and driving off silicon from the silicon carbide, this silicon reacted with CO vapors entrapped in the porous mass of carborundum powder according to equation (2). The silica then formed could react

according to equation (1) and so on. Accordingly another experiment was tried using a rod 10 mm. (0.4 inch) in diameter and 150 mm. (6 inches) long with a current of 250 amperes. This rod would, of course, be at a much lower temperature than the 6 mm. rod and it was actually found that no decomposition of the silicon carbide occurred, but the recrystallization was satisfactory.

The conclusion to be drawn is that in explaining the phenomena in a carborundum furnace it is better to work on the hypothesis that we have appreciable vapor pressures not only of silica and silicon but of carbon and silicon carbide. In any case before that hypothesis ceases to be useful we need more experimental evidence than has so far been disclosed.

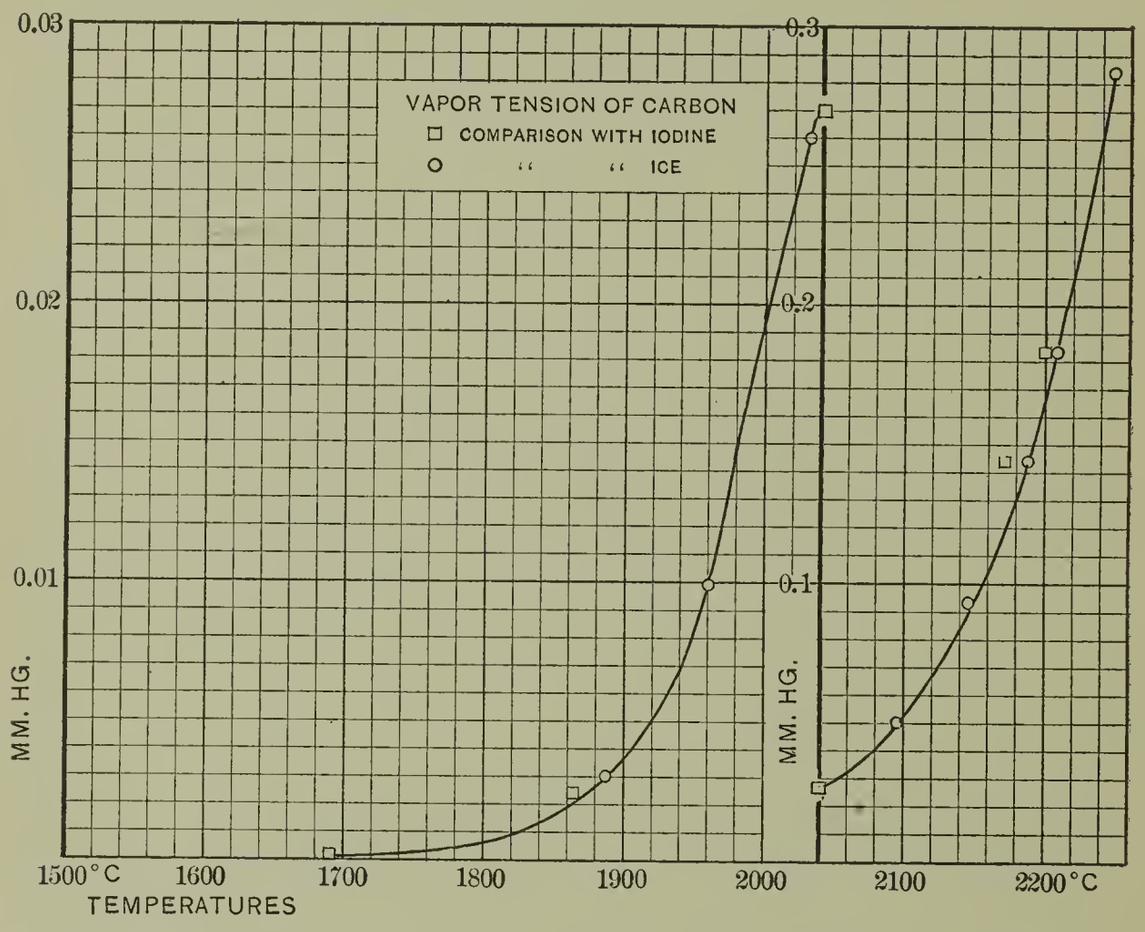
W. R. MOTT: The sublimation point of carbon at atmospheric pressure is usually given as 3600° C. (Violle, Comptes rendu, 120, 868, 1895). For silicon, I imagine the boiling point is of the order of 1500° C. (See von Wartenburg, Z. f. anorg. Chem., 79, 76, 1912). The sublimation point of silicon carbide is unknown, but could be near 2200° C. Silicon carbide is assumed to be always dissociated before reaching a vapor stage. I suggest that this assumption needs further investigation, especially if we introduce an excess of one of the products of reaction—silicon vapor which would tend, by the mass law, to hold back the dissociation of silicon carbide vapor. The precipitation of silicon carbide in the cooler parts from its own vapor would explain recrystallization.

The vapor pressure of carbon at 2200° C. is extremely small, but quite measurable, as shown by the miligrams of carbon soot on the globes of incandescent carbon lamps after burning several hundred hours. However, we do not know the rate of diffusion of carbon vapor but we do know that some extremely small vapor pressures can show very marked properties, as demonstrated by Sir Humphrey Davy over a century ago. (He used a method of calculating vapor pressures still of some value. See Works of Sir Humphrey Davy, Vol. VI, p. 251, On Electrical Phenomena Exhibited in Vacuo.)

I recall that Mr. FitzGerald used kerosene in one of his experiments. Would not the volatile hydrocarbons play an important part?

W. C. Moore: With reference to Mr. FitzGerald's experiments in which he packed silicon in a graphitized tube—possibly the graphite itself may be porous enough to hold considerable quantities of air.

Jos. W. Richards: I think we have sufficient ground for calculating a vapor tension curve of carbon with pretty fair probability as to its reliability. Drawing a vapor tension curve of carbon with an assumed normal vaporizing point of 3600° C., at 760 mm. pressure, and using for comparison the known vapor tensions of iodine and of ice, we have the following diagram:



This shows that carbon has a probable vapor tension of

0.001	mm.	of	Mercury	at 1820° C.
0.005	"	"	"	" 1920° C.
0.01	"	"	"	" 1960° C.
0.02	"	"	"	" 2000° C.
0.03	"	"	"	" 2060° C.
0.05	"	"	"	" 2100° C.
0.10	"	"	"	" 2155° C.
0.20	"	"	"	" 2215° C.
0.30	"	"	"	" 2255° C.

When it is remembered that the Sherardizing process of coating iron with zinc operates at vapor tensions of the zinc from 0.02 mm. Hg to 0.10 mm., there seems ample ground for assuming that in the carborundum furnace, with temperatures up to 2200°, and vapor of carbon present up to 0.2 mm. Hg pressure, that the carbon vapor can probably play an important part in the chemical reactions taking place in the furnace.

A. T. HINCKLEY: I would like to ask Mr. Tone how the temperature measurements given in the paper were made. It makes a difference, in measuring temperatures, what the vapor is in the space, if you use an optical method. If it were a direct radiation method, it makes a difference what the surface is.

F. J. TONE:. The theory I have offered for the formation of carborundum is based, of course, on the assumption that carbon has no appreciable vapor tension at the temperature of the furnace. If it has such vapor tension, the theory fails.

Sir William Crookes has made some calculations on the vapor tension of carbon, and the results are given in the proceedings of the Royal Society of London, Section A, 1905. Making certain assumptions he calculated that carbon had a zero vapor tension at 3000° absolute, or 2700° C. Aside from this, the experimental evidences, I think, are very important. I have mentioned the graphite pseudomorphs of carborundum. They retain not only their crystalline outline, but the markings, workings and spirals that occur on the faces of the crystals, often 40,000 to 50,000 to the lineal inch. I believe that if the vapor tension of carbon plays an important part in the furnace, it must be of considerable magnitude and in such case the outlines and markings would disappear. I might also cite the Acheson furnace for graphitizing carbons. The graphitized carbons retain their outline, not being corroded or eaten away, in any case, except where in contact with silica or with the mixture used in packing the furnace.

The main criticism I would make of Mr. FitzGerald's experiments is that they are not carried out in a way by which silicon or silica or oxygen are absolutely excluded. A very small amount of air or silica is sufficient to satisfy my equation (2). Theoretically, an infinitesimal amount of silica or oxygen would recrystallize a very large amount of carborundum. In Mr. FitzGerald's

second experiment in which a mass of pulverized silicon surrounds the rod, this mass will certainly be pervious to air. I am also not prepared to say what rôle carbon dissolved in molten silicon might play in the result he obtained. The same thing is true of the charge inside the carbon tube in experiment 3; the carbon tube might be very pervious to gases, and the silicon, if vaporized, would react with the carbon monoxide to form recrystallized carborundum in accordance with equation (2).

Regarding the recrystallization of carborundum, results based on a large amount of experimental work have shown us that if a molded form of carborundum powder is placed in a mixture of silica and carbon in the carborundum furnace, the recrystallization is very satisfactory. The crystals are large and the mass has good mechanical strength and tenacity. If the same article, composed of carborundum powder, is placed in fire sand which is much lower in silica, the recrystallization is less satisfactory, the crystals are small and the structure is weak. If we continue further and place the same article in a packing of pure carborundum, the recrystallization is still more unsatisfactory and the article has no mechanical strength. We have found that the degree of recrystallization is a direct function of the concentration of silicon and silica vapors in the furnace. To avoid the errors which might creep into Mr. FitzGerald's experiment on recrystallization, our laboratory repeated the experiment in an Arsem furnace, which is a closed type of vacuum furnace. We used a reduced atmosphere of carbon monoxide. The crucible of the furnace was filled with 3-F powdered carborundum, carefully treated with sulphuric, nitric and hydrofluoric acids, so that all silica or silicon was removed. The temperature of the crucible here shown has not been uniform throughout its length. In the upper or hottest zone the SiC powder is completely converted to graphite. In the next lower zone are found large crystals of carborundum or recrystallized carborundum. In the lowest zone the powder is unchanged. From a casual inspection one might say that the recrystallization proves FitzGerald's theory, but closer examination shows that the graphite is very fine and consists of pseudomorphs, not of large crystals of the adjacent zone, but of the fine powder of the lower zone. This shows that the large crystals were formed only after a silicon atmosphere had

been produced from a disassociation of the carborundum powder. I think if Mr. FitzGerald's experiments were repeated in a closed atmosphere, the results would be entirely different.

My statement of temperatures of the respective zones of the furnace are based on determinations given in papers by H. W. Gillett and L. E. Saunders.[1]

HORACE W. GILLETT: We have tried the silfrax tubes as protection for pyrometers for use in molten brass and bronze. The tubes withstand plunging, cold, into the molten metal without cracking, and withstand oxidation remarkably well.

However, base metal couples deteriorate rapidly when in direct contact with the silfrax, and the time lag in a silfrax tube thick enough to be mechanically strong, is too great for our purpose. By using a nickel tube with a tiny molybdenum tube on its end, and a silfrax tube about the nickel tube, with the molybdenum projecting through the silfrax so that only silfrax and molybdenum come in contact with the metal, a useful protecting tube is obtained. The silfrax is an essential part of the outfit, and we feel grateful to Mr. Tone for making this material available.

The silfrax tubes are too permeable to gas, and to zinc vapor, when used on brass at high temperatures, to allow the satisfactory use of optical pyrometers. An impervious glaze on the inside of the tube, which I understand Mr. Tone is trying to develop, is needed.

W. C. MOORE: I ask Mr. Tone if he has kept any of graphite pseudomorphs at a constant high temperature for a long time to see if they finally lose their shape?

F. J. TONE: Only at the temperature of the carborundum furnace, at the outside of the core, which is 2240° C., and for the regular furnace period of 36 hours.

F. A. LIDBURY: The fact that the pseudomorphs do not lose their shape would not be inconsistent with the existence of a fair vapor tension if they remain, as seems possible, in contact with gases saturated with carbon vapor, such as might be imagined to be continually supplied by the core.

JOS. W. RICHARDS: I have seen a number of these pseudo-

[1] Jour. Phys. Chem., **15**, 213; and L. E. Saunders, Trans. Amer. Electrochem. Soc., **21**, 425.

morphs of the graphite, and while they have the same general form and approximately the form of the carbon crystals, they lack entirely, as far as I have seen, the sharpness of outline of the carborundum crystal. They have a slightly round or worn appearance. Perhaps Mr. Tone has seen others as sharp as carborundum crystals, but I have not.

F. J. Tone: Some discussion might have been avoided if I had defined my use of the term "appreciable vapor tension." I do not contend that the vapor tension is not calculable or measurable, but that it is not sufficient to be considered as a practical factor in the formation of carborundum and in explaining the various phenomena of the furnace such as the formation of large crystals, recrystallization, the local disappearance of carborundum and the stability of the graphite pseudomorphs.

A paper presented at the Twenty-sixth General Meeting of the American Electro-
1, 1914, President F. A. Lidbury in the Chair.

THE CONSTANCY OF BASE METAL THERMOCOUPLES AS RELATED TO THE MICROSTRUCTURE

By O. L. Kowalke.

In a previous paper on base metal thermocouples[1] it was shown that some couples retained a reasonably constant calibration during a series of tests, while others did not. The reasons for deviations from the original calibrations did not appear to be due entirely to mechanical strains or lack of uniformity in the metal. It seemed probable that there might have been changes in the structure of the metal due to heat treatments, or that such alloys were used in the wires which when exposed to high temperatures would produce heterogeniety. The purpose of this investigation was to establish relations between the microstructures of the wires of the couples and their calibrations under various conditions of test.

The assistance of Messrs. J. H. Wolfe and R. G. Waltenberg in obtaining these data is gratefully acknowledged.

PROCEDURE.

Five separate samples about one-quarter of an inch long were cut from each of the wires of the couples. Sections of each set of samples were prepared for microscopic examination to show the original structure and that following a two-hour heat treatment at 400°C., 600°C., 800°C., and 1,000°C. respectively.

The heat treatment of the samples was made in the following manner. A complete set of samples from each couple was mounted on an asbestos board and put into a muffle 3¾″ x 3¾″ x 2″ high. The muffle was wound with resistance wire and heated electrically. The temperature in the muffle was measured by a

[1] Trans. Am. Electrochem. Soc., Vol. XXIV, 1913, p. 377.

calibrated thermocouple indicating on a Keiser & Schmidt millivoltmeter, and was maintained constant at the desired point for a period of two hours. The samples were then removed from the furnace and cooled in the air.

After cooling they were polished to a mirror surface on a section perpendicular to the axis of the wire and mounted on a glass slide in the usual manner.

The samples were first examined to determine whether any structure was brought out by polishing, as would be the case with a mixture of hard and soft metals. Various etching agents were then tried. It appeared that solutions of 5 percent picric acid in alcohol, and 5 percent iodine in alcohol were the two etching agents that brought out the structures best. As soon as a good structure was obtained a photomicrograph was taken. The structures are shown on the accompanying plates.

COMPOSITION OF THE WIRES.

The wires of these couples were of sizes ranging from one-eighth inch to one-sixteenth inch (3.2 mm. to 1.6 mm.) diameter. A quantitative analysis of each wire showed the following:

	Positive Terminal		Negative Terminal	
Couple No. 6A.....	Ni	86.0	Ni	94.6
	Cr	14.3	Fe	2.2
		100.3	Si	2.4
				99.2
Couple No. 7A.....	Cr	10.01	Al	3.30
	Fe	30.00	Ni	96.4
	Ni	59.9		99.7
		99.91		
Couple No. 18.....	Fe	99.9	Al	1.14
			Ni	98.3
				99.44
Couple No. 20.....	Fe	99.8	Cu	52.3
			Ni	48.0
				100.3
Couple No. 21.....	Cr	16.8	Fe	1.08
	Ni	83.3	Ni	98.90
		100.1		99.98

OBSERVATIONS.

Couple No. 6A. After being heated for 24 hours at 800°C. this couple showed a departure of 125°C. at 35 millivolts from the original calibration. Inspection of the micrographs of the negative terminal shows in the dendrite structure a distinct segregation of material. These dendrites are due apparently to the presence of silicon. Binary alloys of nickel and silicon show this segregation phenomenon, while nickel iron alloys, low in iron, show a uniform structure indicating a solid solution. The micrographs of the positive terminal show the condition of a solid solution.

In some subsequent tests on a negative terminal of another couple, represented to be like the one tested, in which a break occurred, the following phenomena were observed. A longitudinal section at the point of rupture showed crystal grains and segregations about four times as large as those obtained at a point where the wire had not been exposed to the high temperatures.

It would appear, then, that the cause for the large change in calibration was due to the segregation of the material in the negative terminal and that silicon is a metal of doubtful value in thermocouple alloys.

Couple No. 7A. The departure from the original calibration to the last was gradual and increased from zero to about 25°C. at 24 millivolts. In the micrographs of both negative and positive terminals, the structure of solid solution appears throughout the tests. In the treatment at 600°C. for the negative terminal there appears to be a suggestion of crystal structure, but no change in the calibration is apparent.

Couple No. 18. The big change in calibration for this couple occurred between the initial calibration with 4 inches heated and the second calibration with 15 inches heated. This is due, no doubt, to mechanical strains. After the first heat treatment the change in calibration is no greater than other couples, being only about 30°C.

The micrographs of the negative and positive terminals show a constancy of structure throughout the treatments except for that at 1,000°C. where a large crystal structure appears in the

iron.' The nickel-aluminium alloy of the negative wire does not change from the characteristics of a solid solution.

Couple No. 20. A change in calibration of only about 20°C. was observed over the entire range. The micrograph of the negative terminal shows that here again the solid solution of nickel in copper has maintained constant calibration. The micrograph of the positive terminal of iron shows characteristic structures for the various temperatures.

It would seem fair to predict that "constantan," an alloy of 60 percent copper and 40 percent nickel, would give equally good results as the above alloys of copper and nickel.

Couple No. 21. This couple was not reported in the first paper, but gave results in calibration that were similar to those of Couple No. 7A. Couple No. 21 had for its positive wire an alloy known to the trade as "Excello," and the negative wire was known as "Pure Nickel." Both of these wires were purchased originally as resistance wire and were made into couples in this laboratory.

The micrographs of both wires show conditions of solid solutions in all cases.

CONCLUSIONS.

From these data it would appear reasonable to conclude that those metals which form solid solutions with one another, give the best results, providing that the thermoelectric force is satisfactory and the melting point high.

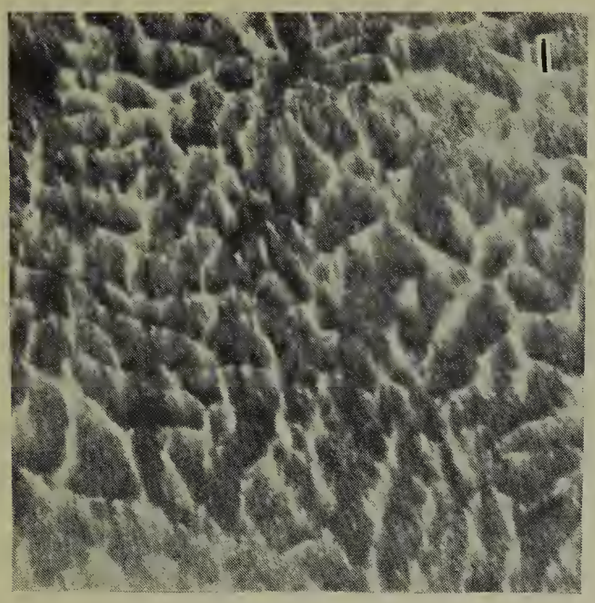
Silicon does not appear to be a desirable constituent in a nickel alloy for thermocouple material.

Any segregation of the metal has an undesirable effect on the constancy of the couple.

Wires containing but one metal showed a satisfactory structure.

Chemical Engineering Laboratory,
University of Wisconsin.

Couple No. 6A. Positive Terminal.
Etched with Iodine. Ni = 86.0%; Cr = 14.3%.



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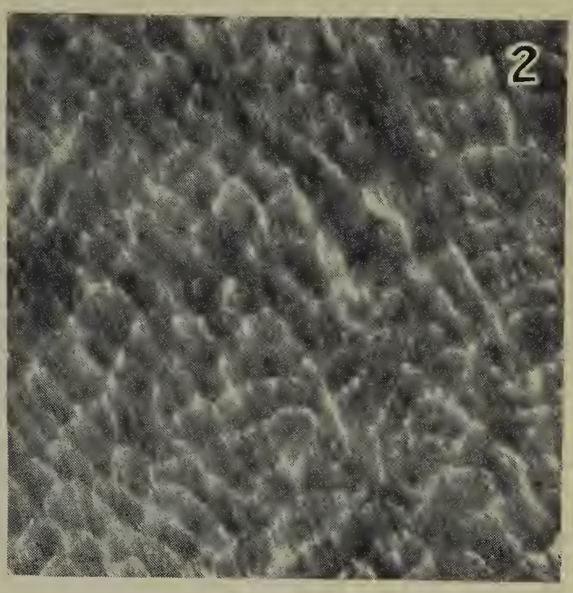

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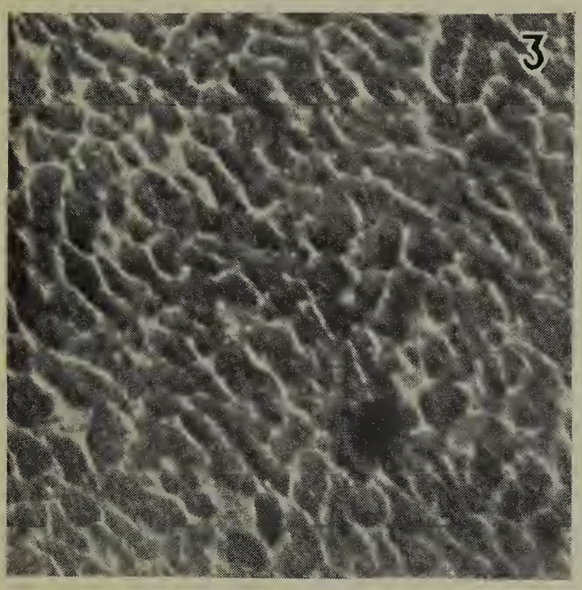

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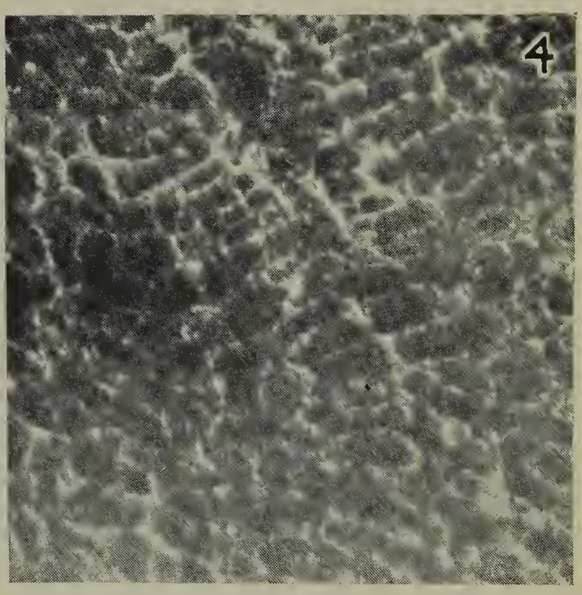

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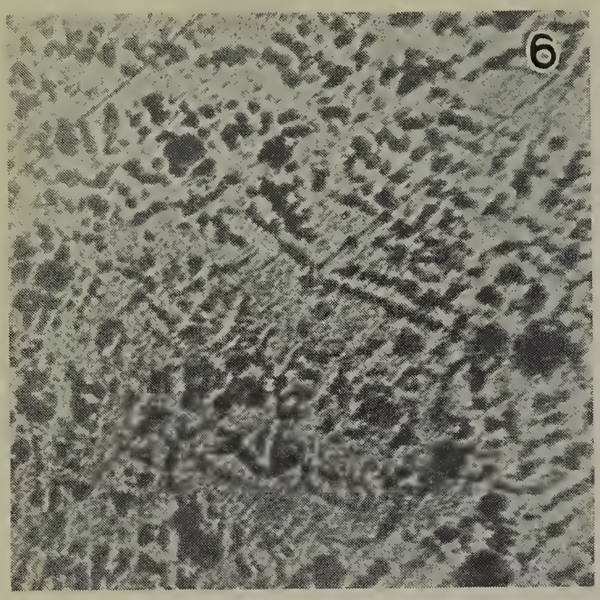
FIG. 1. Original Structure.
FIG. 2. Heated at 400° C.
FIG. 3. Heated at 600° C.
FIG. 4. Heated at 800° C.
FIG. 5. Heated at 1000° C.

Couple No. 6A. Negative Terminal.
Etched with Iodine. Ni = 94.6%; Fe = 2.2%; Si = 2.4%.



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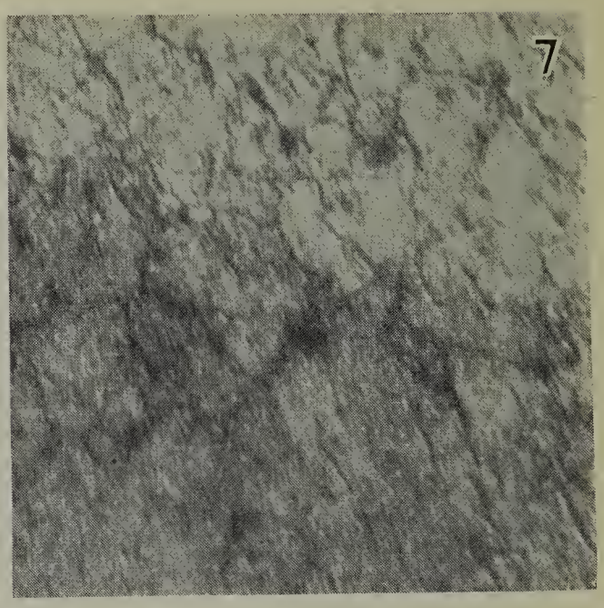

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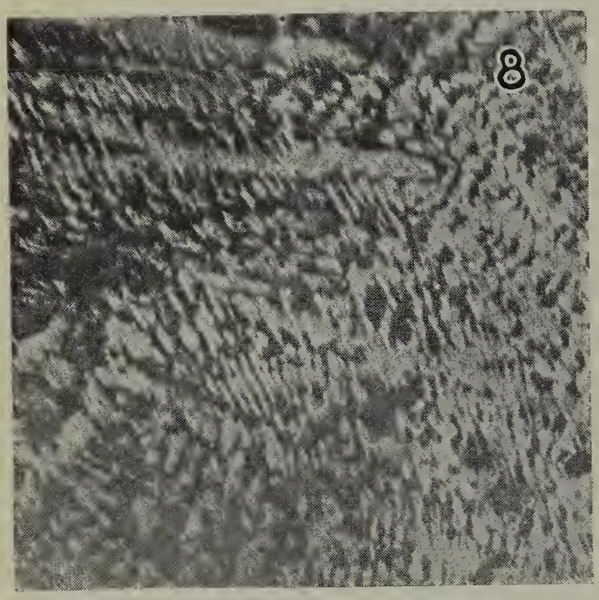

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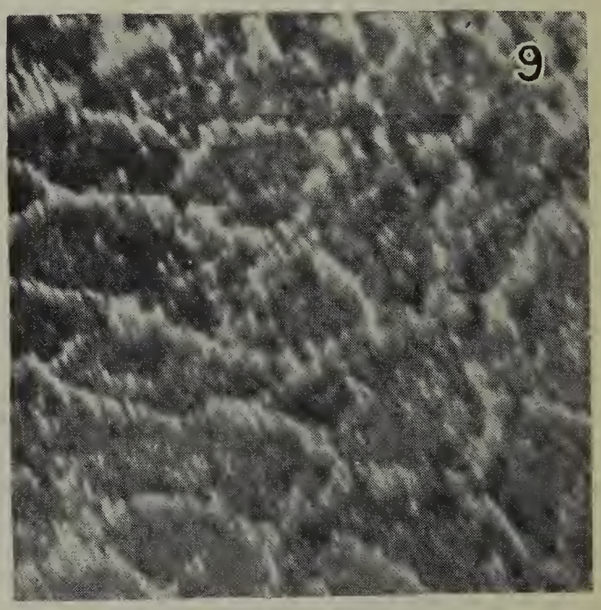

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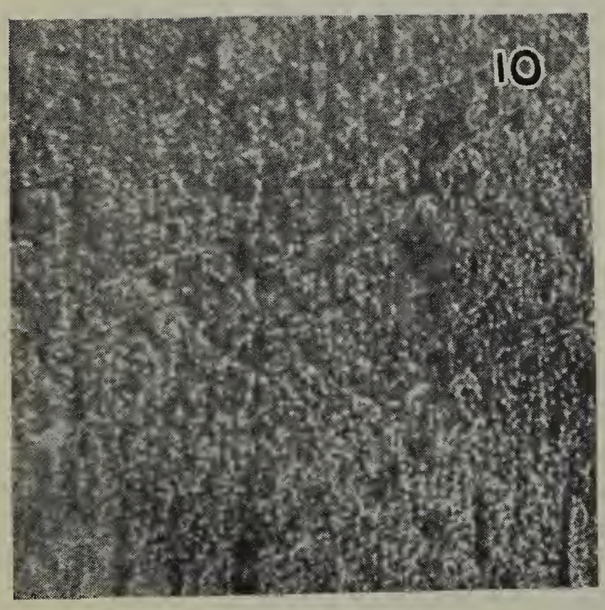

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FIG. 6. Original Structure.
FIG. 7. Heated at 400° C.
FIG. 8. Heated at 600° C.
FIG. 9. Heated at 800° C.
FIG. 10. Heated at 1000° C.

Couple No. 7A. Positive Terminal.
Etched with Iodine. Cr = 10.01%; Fe = 30.00%; Ni = 59.90%.

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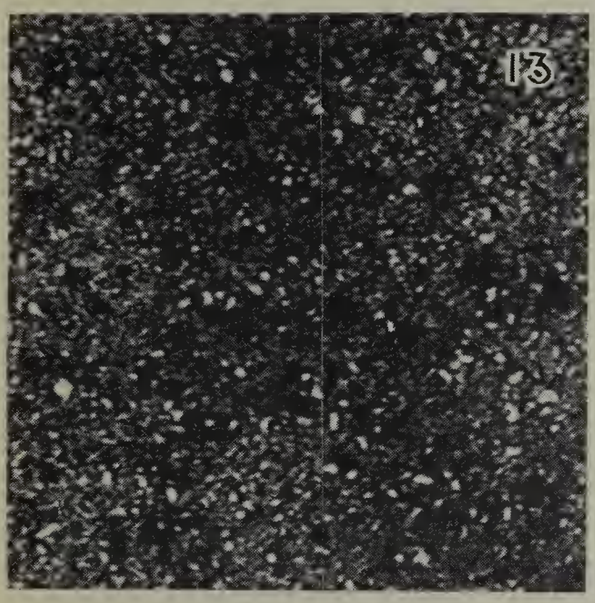

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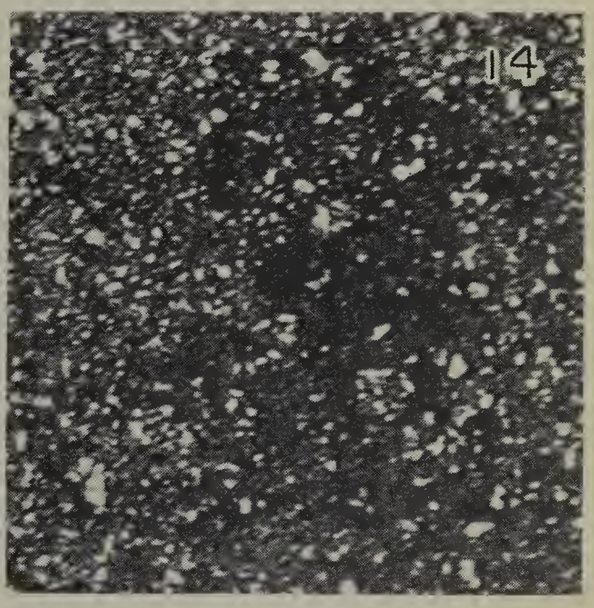

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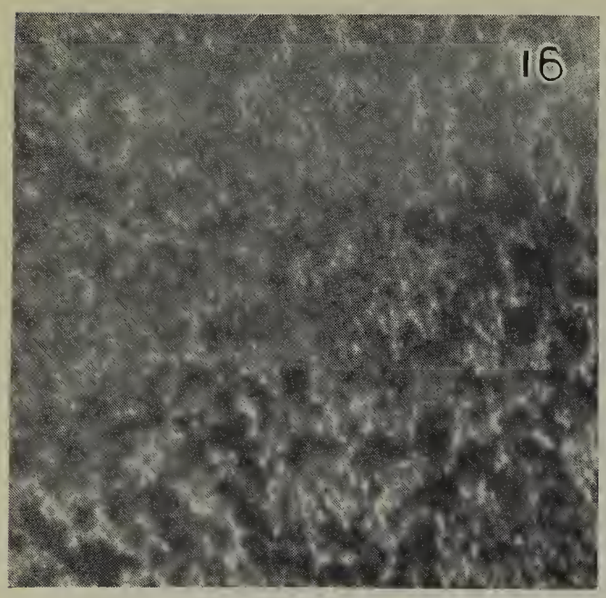
FIG. 11. Original Structure.
FIG. 12. Heated at 400° C.
FIG. 13. Heated at 600° C.
FIG. 14. Heated at 800° C.
FIG. 15. Heated at 1000° C.

Couple No. 7A. Negative Terminal.
Etched with Iodine. Al = 3.3%; Ni = 96.4%.



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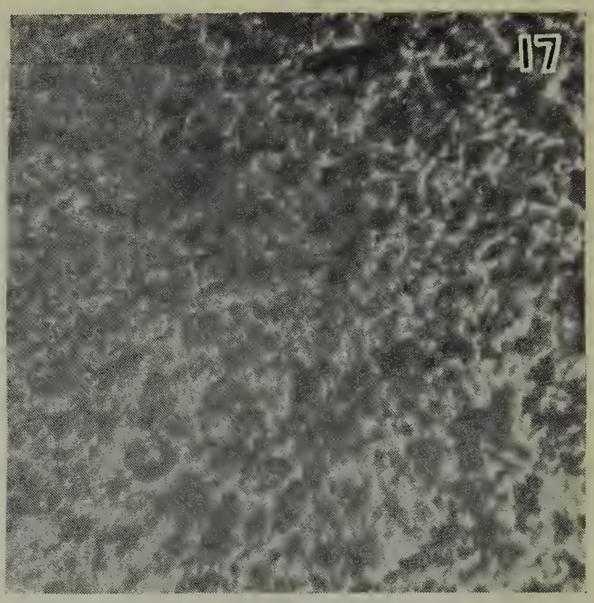

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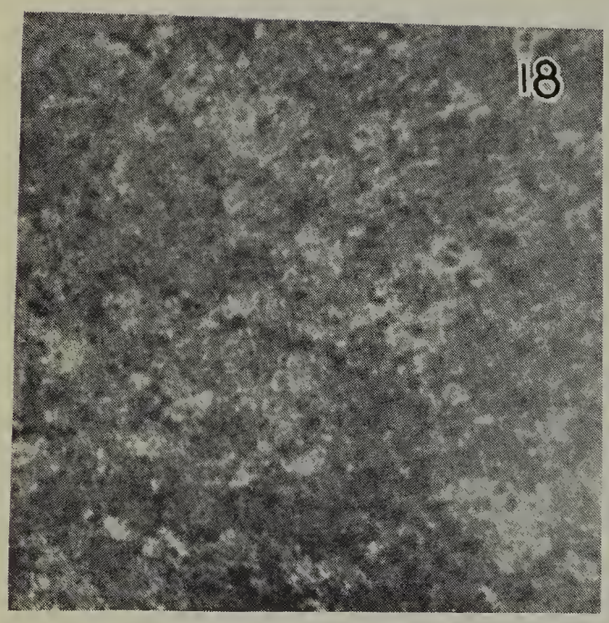

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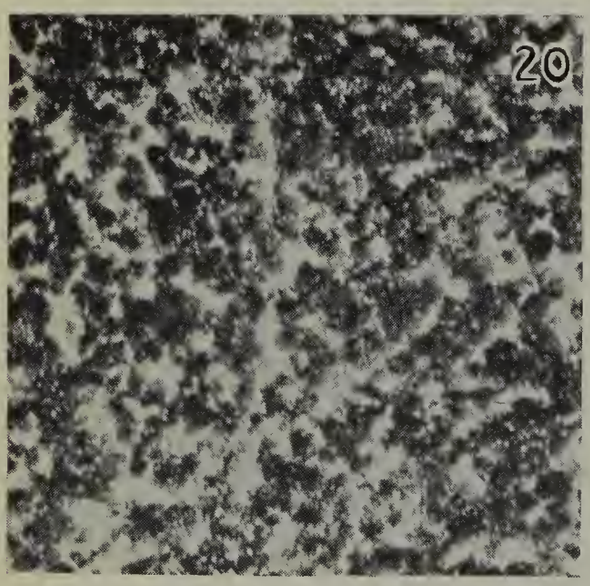

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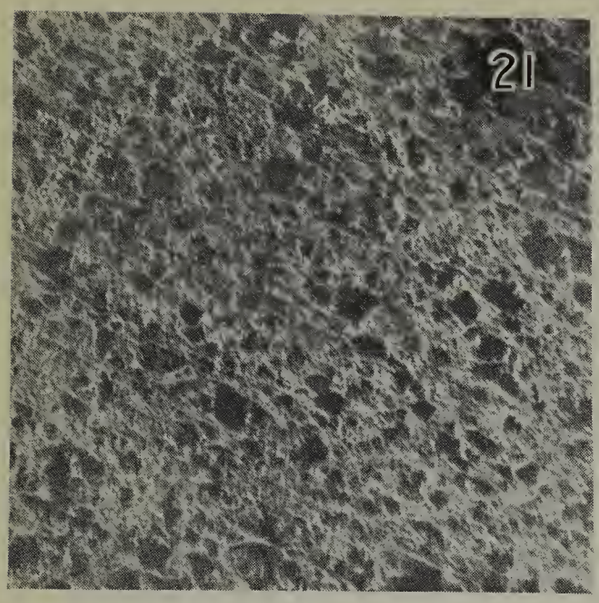
FIG. 16. Original Structure.
FIG. 17. Heated at 400° C.
FIG. 18. Heated at 600° C.
FIG. 19. Heated at 800° C.
FIG. 20. Heated at 1000° C.

Couple No. 18. Positive Terminal.
Etched with Iodine. Fe = 99.9%.



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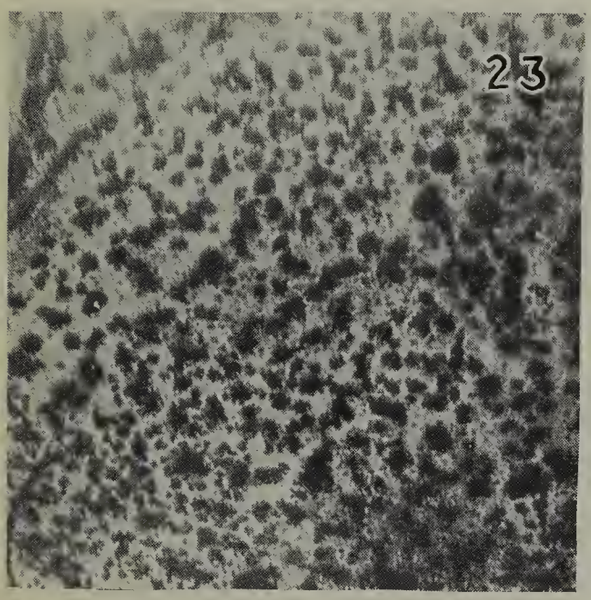

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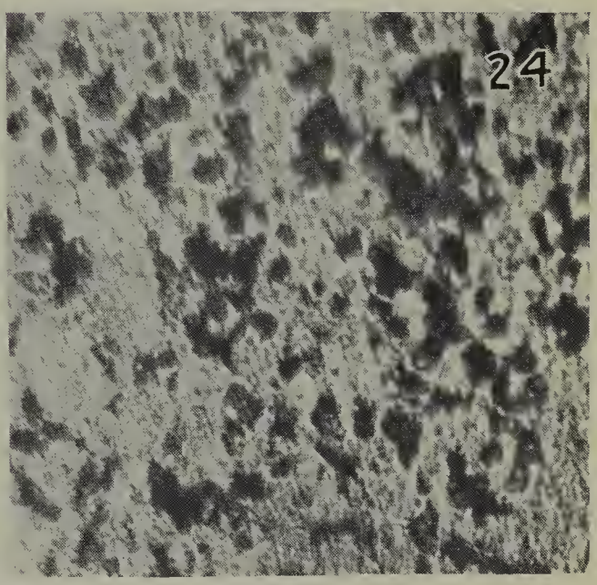

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FIG. 21. Original Structure.
FIG. 22. Heated at 400° C.
FIG. 23. Heated at 600° C.
FIG. 24. Heated at 800° C.
FIG. 25. Heated at 1000° C.

Couple No. 18. Negative Terminal.
Etched with Iodine. Al = 1.14%; Ni = 98.3%.

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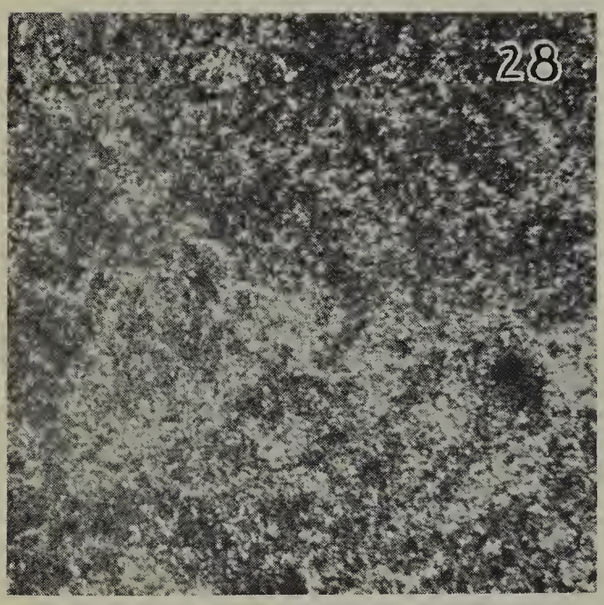
× 80



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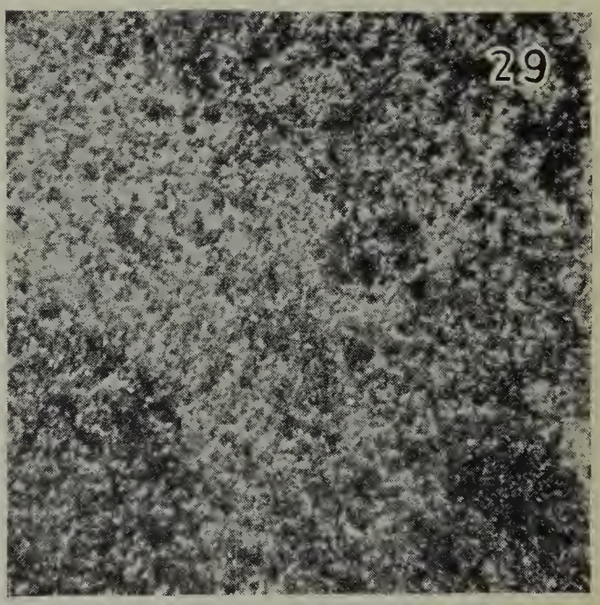

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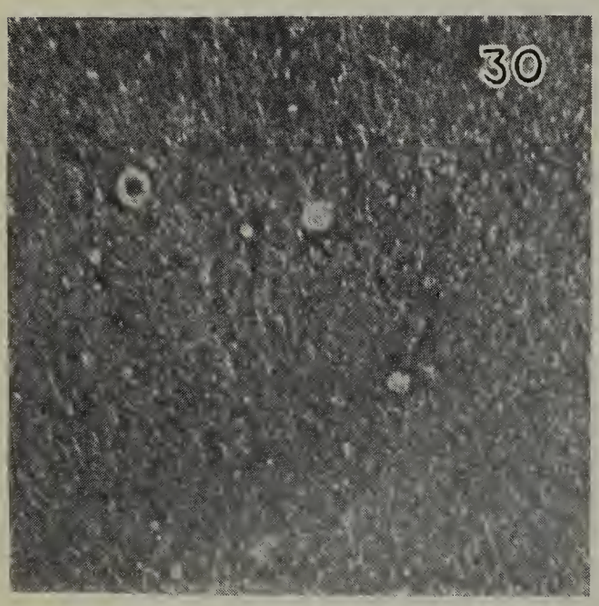

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FIG. 26. Original Structure.
FIG. 27. Heated at 400° C.
FIG. 28. Heated at 600° C.
FIG. 29. Heated at 800° C.
FIG. 30. Heated at 1000° C.

Couple No. 20. Positive Terminal.
Etched with Picric Acid. Fe = 99.80%.

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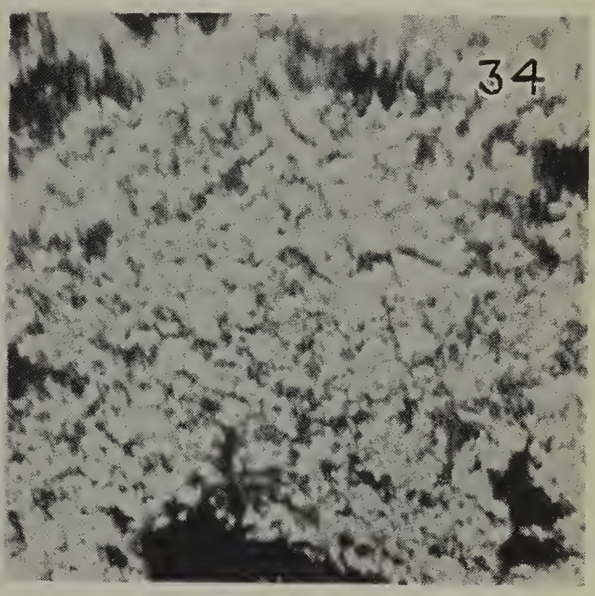
× 225

× 225



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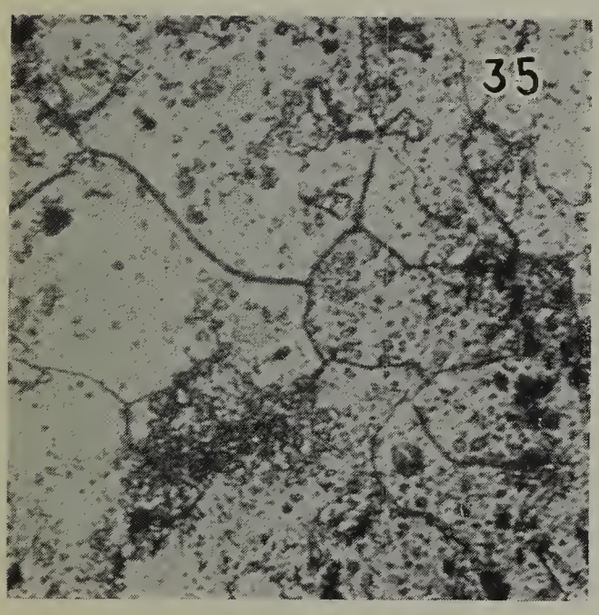

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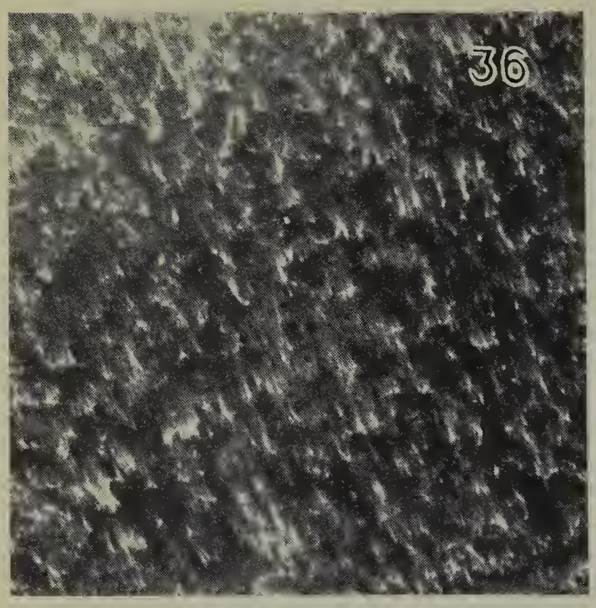
FIG. 31. Original Structure.
FIG. 32. Heated at 400° C.
FIG. 33. Heated at 600° C.
FIG. 34. Heated at 800° C.
FIG. 35. Heated at 1000° C.

Couple No. 20. Negative Terminal.
Etched with Iodine. Cu = 52.3%; Ni. = 48.0%.



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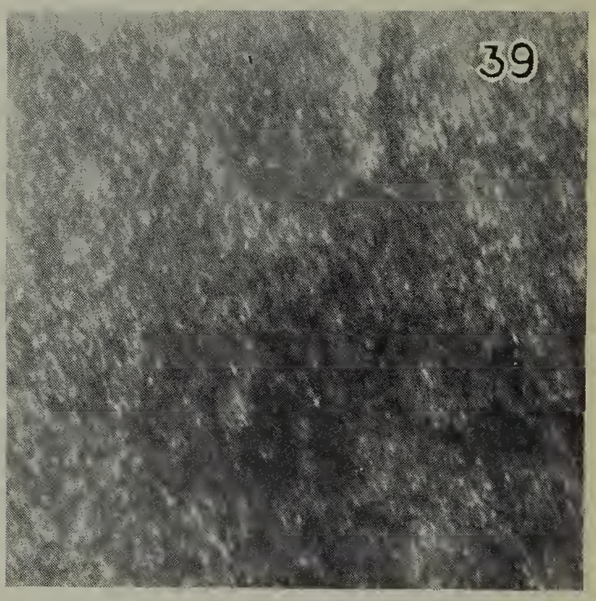

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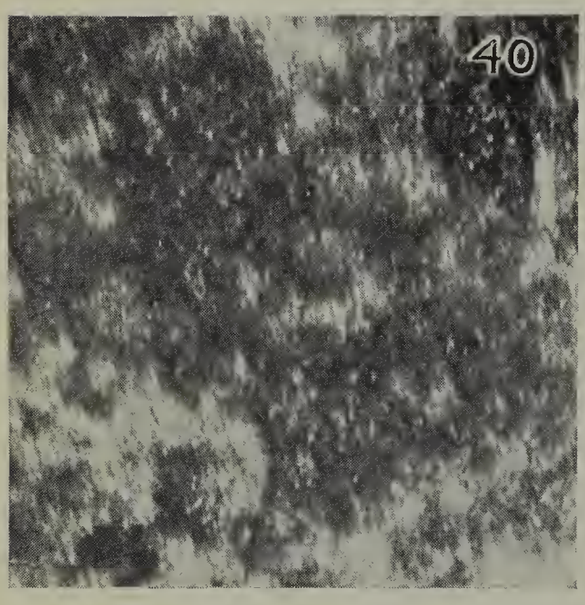

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FIG. 36. Original Structure.
FIG. 37. Heated at 400° C.
FIG. 38. Heated at 600° C.
FIG. 39. Heated at 800° C.
FIG. 40. Heated at 1000° C.

Couple No. 21. Negative Terminal.
Etched with Iodine. Fe = 1.08%; Ni = 98.90.%.

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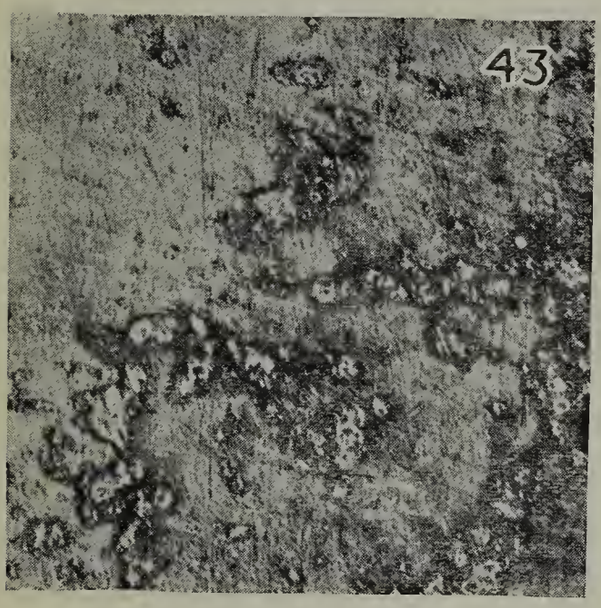
× 80



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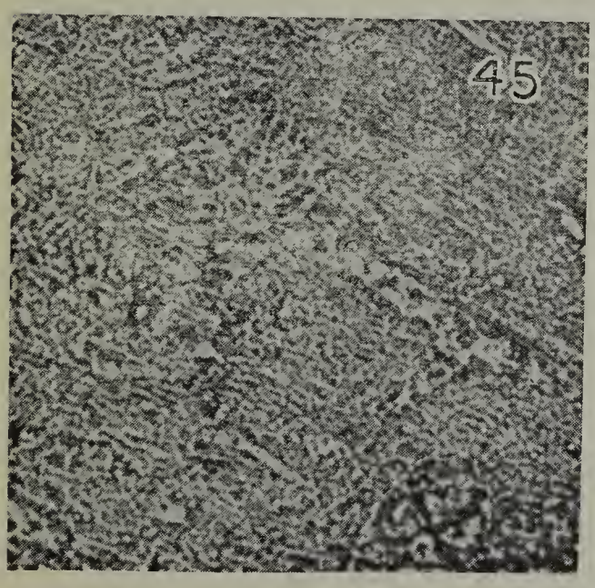

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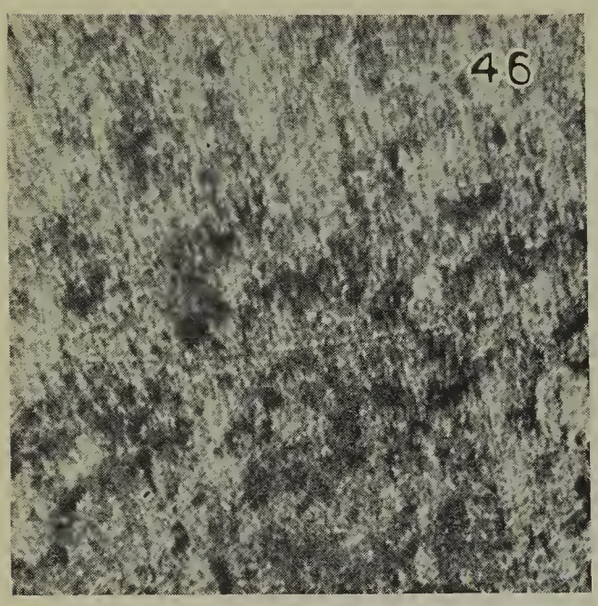
FIG. 41. Original Structure.
FIG. 42. Heated at 400° C.
FIG. 43. Heated at 600° C.
FIG. 44. Heated at 800° C.
FIG. 45. Heated at 1000° C.

Couple No. 21. Positive Terminal.
Etched with Iodine. Cr = 16.8%; Ni = 83.3%.



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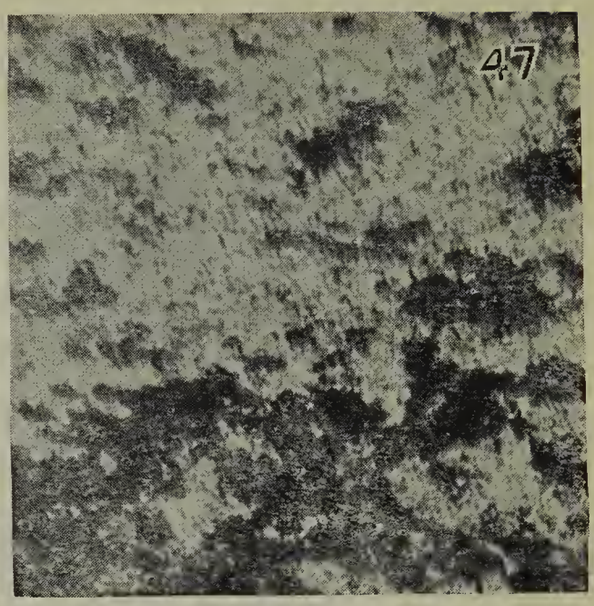

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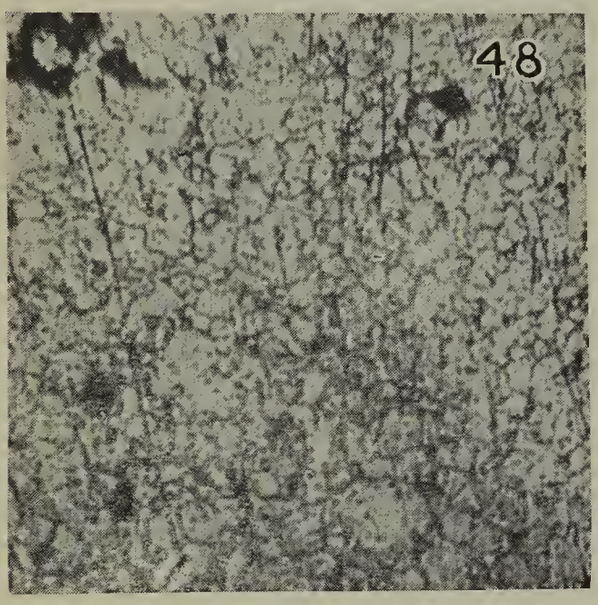

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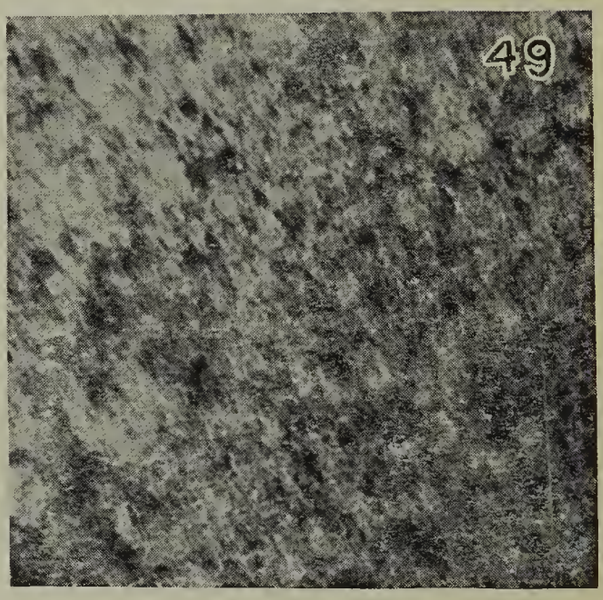

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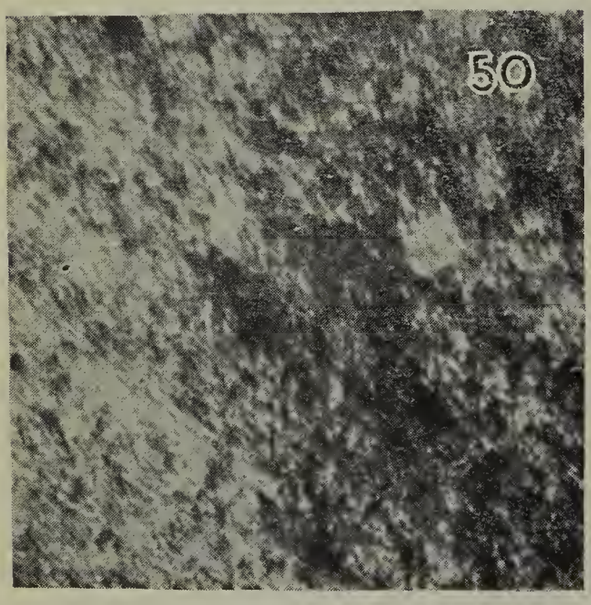

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FIG. 46. Original Structure.
FIG. 47. Heated at 400° C.
FIG. 48. Heated at 600° C.
FIG. 49. Heated at 800° C.
FIG. 50. Heated at 1000° C.

DISCUSSION.

W. R. MOTT: I believe Mr. Tone will bear me out in the statement that freshly made alloys of silicon with iron or manganese, frequently recrystallize shortly ofter being taken out of the furnace.

Silicon is interesting in another way, as regards its thermoelectric characteristics. Messrs. F. Fischer, R. Lepius and E. Boerwind have reported on the thermoelectric power of silicon.[2] If it is deoxidized, the e. m. f. is exactly in the reverse direction from silicon which contains dissolved silica, and the difference amounts to almost 1000 microvolts per degree.

CHARLES B. THWING (*Communicated*): I for one am grateful to Professor Kowalke for carrying his researches on thermocouples to so interesting a conclusion. While seeking a relation between thermo-electromotive force and co-efficient of expansion, some ten years ago, I met some striking exhibitions of the effect of aggregation of crystals as changed by heat upon both of these properties of certain metals and alloys.

Antimony, for example, when cast in an iron chill crystallized radially. With repeated heating to 100 degrees, the length continued to increase and the electromotive force to diminish for several successive heatings. Since the electromotive force differs for different axes, I attributed the change to a re-arrangement of the crystals in which some crystals were oriented more nearly parallel to the axes, or as they would have arranged themselves if the metal had cooled more slowly when cast. The change of electromotive force on the first heating was more than ten percent.

I hope that Professor Kowalke will apply the same tests to platinum thermocouples that have been damaged by long use. Such couples may often be restored by glowing them with a current not sufficient to melt the wire but to reach, say 1500 degrees. Microphotographs taken before and after such a restoration would be of exceeding interest.

JOS. W. RICHARDS: Some of the so-called standard couples which have been standardized, and probably accurately standardized, in the laboratory of the maker, when they have been brought into the works and used again, will show a variation of from

[2] Physik. Zeit., **14**, 439-46; Zeit. anorg. Chem., **81**, 243-65.

10° to over 100° C. from the standardization. The changing micro-structure is probably the cause of this great deviation, which makes them almost unusable for accurate work.

B. L. GLASCOCK: The couples that come from the maker direct to the user generally are not calibrated by the user, who assumes that the calibration of the maker is correct. Four couples supposed to have been standardized by the manufacturer, when they were checked against a platinum—platinum-rhodium couple, were found to vary from 8° to 52° at a temperature of 1000°. We have had couples which had been used and repaired that varied as much as 100° at 1000° C.

M. G. LLOYD: I think it would add a great deal to the value of this information if the last two speakers would say what the materials of the couples were.

W. A. MCKNIGHT: The makers of the couples on which these tests were made will not state the composition of the alloy used and no analyses have been made which would tend to show the composition of the couple. However, it is one that is manufactured for use at 1250° C., and at 1000° C. the conditions stated by Mr. Glascock were found to exist.

A paper presented at the Twenty-sixth General Meeting of the American Electrochemical Society, at Niagara Falls, October 3, 1914, President F. A. Lidbury in the Chair.

THE EVAPORATOR AND THE POWER PROBLEM IN ELECTROCHEMICAL PLANTS.

By Otto Mantius.

The electrolysis of sodium or potassium chloride solutions will produce liquors which with very few exceptions cannot be used or marketed without further concentration. In most cases considerable quantities of water have to be removed, and it is generally acknowledged that the cost for fuel, labor, repairs, interest and depreciation to be charged to the evaporating equipment have considerable influence on the rentability of the whole plant.

During the last few months we have heard a great deal about the conditions under which reciprocating engines, steam turbines or water power can be used for the generation of electric current to best advantage. In this paper I shall present some of the factors and conditions which should be considered when laying out an electrochemical plant where an evaporating system has to work in connection with water power or steam plant.

I shall not attempt to discuss the efficiency of the great variety of cells which are in daily use for the manufacture of caustic liquors, as the figures given below can readily be changed and discounted for any particular system. I shall not go into the question whether water, gas or steam should be used to produce the electric current. I shall restrict myself entirely to the discussion of those features and conditions which have a bearing on the design of the evaporating equipment.

The installation of an evaporator would be a very simple matter if we had to deal with sugar juice, black liquor or packing-house tank water. A double or triple effect for small quantities and a quadruple effect for larger amounts of liquor could be bought, installed and operated at comparatively low first cost and running expenses. The problem of a caustic liquor evaporator is somewhat complicated by the incrustations of the tubes, the

corrosive action of the liquor itself and the high boiling point of the more concentrated solution, which practically eliminates the use of a quadruple effect even for large quantities, restricts the operation of a triple effect to certain favorable conditions, and makes a double effect the most economical equipment for a plant of medium capacity.

Inasmuch as this statement is quite contrary to ordinary practice, I give the following figures for steam consumption of various equipments, which will explain and prove my contention:

My calculation has been based on the following assumptions:

Capacity in 24 hours.....20,000 lb. (9091 kg.) NaOH.
Power required .....1500 E. H. P.
Weak liquor .....20,000 gal. (75,750 liters) with 120 grams NaOH and 100 grams NaCl per liter.
Heavy liquor .....3440 gal. (13,200 liters) with 700 grams NaOH and 16 grams NaCl per liter.
Evaporation .....16,560 gal. (62,650 liters) in 20 hours = 828 gal. (3135 liters) per hour.

The hourly steam consumption, including radiation loss, preheating of the weak liquor from 70° F. (21° C.) and evaporation of the wash water, will be as follows:

Single Effect—7900 lb.; 6000 lb. direct steam, 1900 lb. exhaust from pumps (3590; 2727 and 863 kg.).
Double Effect—5090 lb.; 3690 lb. direct steam, 1400 lb. exhaust from pumps (2312; 1676 and 636 kg.).
Triple Effect—4600 lb.; 3400 lb. direct steam, 1200 lb. exhaust from pumps (2090; 1544 and 546 kg.).
Triple Effect and Single Effect—4300 lb.; 3000 lb. direct steam, 1300 lb. exhaust from pumps (1952; 1362 and 590 kg.).

Single effect and double effect are to be operated with steam at 7 lb. and 20 lb. gauge pressure (0.5 and 1.3 kg. per sq. cm.), triple effect at from 60 lb. to 70 lb. (4 to 4.7 kg. per sq. cm.) and the combination triple effect and single effect with steam at from 7 lb. to 20 lb. (0.5 to 1.3 kg. per sq. cm.). In the latter arrangement the triple effect concentrates the weak liquor to about 400 grams per liter, and the single effect finishes the process to 700 grams NaOH per liter. This arrangement will permit the use of low pressure steam, and should always be used where larger quantities of liquor have to be handled.

The above figures show that the efficiency of the evaporator increases with the number of effects as usual, even if the dif-

ference is not as large as might be expected from general practice. The comparatively high steam consumption of the high pressure straight triple effect is due to the increased radiation losses and the large amount of wash waters to be handled and preheating to be done.

These conditions will change as soon as we use a part or all of the exhaust steam of engines or turbines for the evaporation of the caustic liquors.

Where the electricity for the cells has to be generated in a steam plant, either steam turbines or reciprocating engines may be used and the exhaust utilized in the evaporator. As the quantity of steam required for power purposes is about three times larger than the amount needed for the evaporator, the ordinary method of using all the engine exhaust at about 5 lb. (0.3 kg. per sq. cm.) pressure for the concentration of the caustic liquor can only be applied in very small plants by running the evaporator about seven hours per day and operating the engine during the rest of the time in connection with the condenser attached to the evaporator.

In most cases it is necessary to run a multiple effect evaporator continuously and utilize only a part of the steam, which has entered the engine or turbine at high pressure and is withdrawn at from 20 lb. to 30 lb. (1.3 to 2.0 kg. per sq. cm.). I find that for this kind of work steam turbines are not quite as suitable as reciprocating compound engines, as in a steam turbine a large part of the work is done during the low pressure period. While actual tests on small compound engines show that 10 lb. (4.5 kg.) steam withdrawn from the receiver at 20 lb. (9.1 kg. per sq. cm.) pressure require only from 5 lb. to 6 lb. (2.25 to 2.75 kg.) extra steam at 125 lb. (8.3 kg. per sq. cm.) pressure, the guarantees given for large steam turbines ask for 8 lb. (3.6 kg.) extra steam at 150 lb. (10 kg. per sq. cm.) pressure added for 10 lb. (4.5 kg.) steam withdrawn at 20 lb. (1.3 kg. per sq. cm.) pressure.

To produce 20,000 lb. (9,091 kg.) NaOH in 24 hours the steam plant should develop 1,500 E. H. P. A compound condensing Corliss engine will use 15 lb. (6.8 kg.) steam per E. H. P. per hour and increase its steam consumption 1 lb. (0.45 kg.) for every 2 lb. (0.91 kg.) discharged from the receiver at 20 lb. (9.1 kg.) pressure or higher.

Assuming that 1 ton of coal furnishes 15,000 lb. (6,820 kg.) of dry steam at a cost of $3.00, *i. e.*, 1,000 lb. (454 kg.) of steam for $0.20, the steam and coal consumption of engine and evaporator combined will then be as follows:

Single Effect.

Engine	1500 x 15 x 24 =	540,000 lb. (245,300 kg.) steam
Evaporator	3000 x 20 =	60,000 lb. (27,270 kg.) steam
Pumps	1900 x 20 =	38,000 lb. (17,270 kg.) steam
		638,000 lb. (289,840 kg.) steam

Fuel cost per day = $127.60; per 1000 lb. (454 kg.) NaOH = $6.38.

Double Effect.

Engine	1500 x 15 x 24 =	540,000 lb. (245,300 kg.) steam
Evaporator	1845 x 20 =	36,900 lb. (16,760 kg.) steam
Pumps	1400 x 20 =	28,000 lb. (13,330 kg.) steam
		604,900 lb. (275,390 kg.) steam

Fuel cost per day = $120.98; per 1000 lb. (454 kg.) NaOH = $6.05.

Triple Effect and Single Effect.

Engine	1500 x 15 x 24 =	540,000 lb. (245,300 kg.) steam
Evaporator	1500 x 20 =	30,000 lb. (13,635 kg.) steam
Pumps	1300 x 20 =	26,000 lb. (11,820 kg.) steam
		596,000 lb. (270,755 kg.) steam

Fuel cost per day = $119.20; per 1000 lb. (454 kg.) NaOH = $5.96.

A straight triple effect naturally can not be operated on steam of 20 lb. (1.3 kg. per sq. cm.) pressure, and live steam has to be used altogether.

Triple Effect.

Engine	1500 x 15 x 24 =	540,000 lb. (245,300 kg.) steam
Evaporator	3400 x 20 =	68,000 lb. (30,900 kg.) steam
Pumps	1200 x 20 =	24,000 lb. (10,900 kg.) steam
		632,000 lb. (287,100 kg.) steam

Fuel cost per day = $126.40; per 1000 lb. (454 kg.) NaOH = $6.32.

In all cases evaporators and pumps work only 20 hours per day, while the cells are naturally operated during the full 24 hours.

These figures show that for a production of 3,600 tons caustic per year the cost of fuel is about $2,000.00 higher for a straight triple effect than for a double effect or combination triple and

single effect. Besides, we must not forget that a high pressure triple effect is considerably more expensive in first cost and repairs, and is sometimes difficult and even dangerous to operate. Wherever possible, evaporators for caustic liquors of high density should be operated under vacuum, as it will prevent loss of caustic and reduce the corrosive action of the liquor.

Quite a number of electrochemical plants obtain the necessary power at very low rates from central power plants, but have to install boilers to furnish steam for the evaporators. At first glance it would appear that a high pressure triple effect might be the most economical equipment, but the following figures will prove that as far as cost of power is concerned a different arrangement is preferable. Let us assume that only $22.00 is charged for one E. H. P. per year and that 1,000 lb. (454 kg.) steam cost $0.20 as before.

Straight Triple Effect.

Cost of power	$\frac{1500 \times 22}{365} =$	$ 90.40
Evaporator and pumps	4600 x 20 = 92,000 lb. =	18.40
		$108.80

Therefore 1000 lb. (454 kg.) NaOH = $5.44.

We have seen before that a combination triple and single effect can be operated at low pressure and uses less steam than a straight triple effect. We can therefore first use the high pressure steam in a special compound engine to furnish a certain amount of power at not any cost for fuel, and then utilize the exhaust for the evaporation of the liquor. Assuming that the engine will use 23 lb. (10.5 kg.) per E. H. P., and therefore produce 130 E. H. P. from the available 3,000 lb. (1,364 kg.), we have only to purchase 1,370 E. H. P. outside power, and the cost will be as follows:

Combination Triple and Single Effect.

Power	$\frac{1370 \times 22}{365} =$	$ 82.60
Engine and evaporator	130 x 23 x 24 = 72,000 =	14.40
Pumps	1300 x 20 = 26,000 =	5.20
		$102.20

1000 lbs. (454 kg.) NaOH = $5.11.

This would mean a saving of over $2,400 per year, less interest, depreciation and repairs of engine. Quite frequently it might not be desirable to install such an engine, but there will always be a number of pumps and small engines the exhaust of which should be used in the evaporating equipment.

We could even go a step further and install a 500 E. H. P. compound condensing engine, use in the evaporator steam taken from the receiver and contract for only 1,000 E. H. P. to be taken from the central station. Considering the cost of extra labor, repairs and depreciation, we would not save much, but the engine could be arranged so as to take up the peak load, for which otherwise in most cases heavy penalties have to be paid, and the plant could be operated at two-thirds of its normal capacity by shutting down the engine without having to pay for more than the 1,000 E. H. P. Besides, the plant has not to be closed down entirely in case the central plant should not be able to furnish the current.

Similar conditions prevail in plants where gas engines or heavy oil engines are used to generate the current for the cells. A double effect or combination triple and single effect using exhaust steam from auxiliary engines and pumps will give the best results.

A type of engine used very little in this country is the Lentz patented superheated steam engine, which even in small units will produce current at surprisingly low cost. I was told last year during a visit to Germany that one E. H. P. can be produced by 10 lb. (4.5 kg.) of steam and less in a 300 kw. unit. This would make the Lentz engine the ideal motive power for small plants furnishing bleach liquor and caustic for paper mills.

The foregoing figures lead to the conclusion that a straight triple effect operated with high pressure steam should never be used for the concentration of caustic liquors. The first cost is high, and the expenses for fuel, repairs and renewal are more than for any other arrangement. Besides, such a triple effect is considered by many people a general nuisance and source of trouble on account of the difficulties in keeping joints and valves tight.

As far as the cost of fuel per 1,000 lb. (454 kg.) NaOH is concerned, there is not much difference between a single, double

or combination triple and single effect, but low first cost and simplicity of operation should induce us to choose a single or double effect for small capacities up to 5 tons NaOH per day; for larger plants, and especially where the cost of coal is high, and large amounts of cooling water can be had at only considerable expense, a double effect or combination triple and single effect should be installed. The proper arrangement has to be determined for each plant, and no general rules can be given.

I wish to add a few words concerning the general design of the evaporating equipment:

1. For small single effects operating only ten hours per day no reserve is needed, as all repairs can be done over night. The piping of multiple effects running continually should always be arranged so that any pan can be cut off entirely for repairs.

2. Steel, charcoal iron or nickel steel tubes should be used only up to 40° Bé., as the corrosive action of caustic up to this density is very small, and any kind of tube will last a long time, unless the liquor contains a considerable amount of corrosive impurities.

3. For the concentration from 40° to 50° Bé. and higher, only special cast iron tubes should be used, because cast iron is the only material that will withstand the action of the hot concentrated caustic liquor. In Germany almost all the caustic liquor above 40° Bé. is handled in cast iron evaporators. The first cost of such an equipment is somewhat higher, but this difference is soon paid for by the saving in repairs and the increased efficiency of the plant, as there is no time lost in shutting down to replace worn-out tubes. Some of the largest manufacturers of caustic prefer to use copper tubes almost exclusively, as copper will wear longer and more uniformly than steel or charcoal iron. Besides, the scrap value of the discarded tubes is quite high. On the other hand, the first cost is considerable, and where large amounts of salt have to be handled the copper tube disappears quickly on account of the mechanical abrasion caused by the salt passing along the copper surface at high speed.

It was not my intention to consider all possible combinations between evaporator and the source of power, as this is not the time and the place to make such an exhaustive investigation. I only wished to point out the economic and practical disadvantage

of a high pressure straight triple effect when handling caustic liquor up to 50° Bé., and the good results that may be obtained from a compound or triplex condensing engine when working in connection with a double effect evaporator or a combination of low pressure triple effect and single effect with a cast iron heating surface.

Buffalo, N. Y.,
April 8, 1914.

DISCUSSION.

A. H. Hooker: There are a few questions I want to ask in reference to the system of evaporation you describe. In the first place, with reference to the corrosion of tubes: There seems to be, for instance, in a type of evaporator having vertical tubes a considerable corrosion of these tubes as you increase the concentration of the caustic, and I note that the author speaks of "cast-iron effects" for the higher concentrations. I was wondering whether he considered the corrosion as due to the direct effect of caustic soda on steel or charcoal-iron tubes, or as an abrasive effect, and if so, why the effect of corrosion should be shown in the upper part of the tube rather than uniformly throughout its length; and if the effect was due to corrosion, why there should not be a greater corrosion, for instance, in the first pan of a double effect than in the second pan, since there is a much larger amount of salt separated and handled in the earlier part of the operation. Also, my experience has been that where you have a set of double effect pans operating for the first part of the concentration, and then a single effect pan for the final operation, the corrosion in the single effect pan is much greater than in the other two pans; and yet the amount of salt present there is comparatively small.

It has seemed to me that the corrosion of these tubes might well be due to something like a film evaporation taking place at the upper edge of the tube, thus forming a very highly concentrated caustic at the point where the solution broke into steam

bubbles. If cast iron tubes are desirable for highly concentrated caustic, one can appreciate why there should be this wear from the high concentration of small films of caustic at the upper edge of the tube.

There is another question in connection with this system of evaporation that I could not quite understand in going over the figures. This has reference to the caustic liquor evaporated. I notice that you speak of the caustic liquor as containing about 120 grams of NaOH and 100 grams of NaCl per liter. It has occurred to me in looking this over that I have not seen any of the types of cell liquor which would not carry approximately 180 grams of NaCl with 120 grams of NaOH, instead of the 100 grams of NaCl which you mention. This, of course, would nearly double the amount of salt, and this only brings out the question of handling this extra amount of salt and the wash waters which would of necessity be with it. All of which would necessarily call for much larger amounts of water to be evaporated than in the types of liquor which you take for an example.

L. D. Vorce: There are a number of points about the paper which have caused a little question in my mind. In the first place, I do not understand why Mr. Mantius calls for 7 pounds pressure on his single-effect. He is not strictly consistent, for later on in the paper he says 5 pounds. I think he can still cut off a couple more pounds and be about right.

Otto Mantius: The two pounds will not make any difference.

L. D. Vorce: Another point I should have liked to have seen brought out is the combination of the double effect and single effect. He has shown the single effect, the double effect, the triple effect and single effect and triple effect; but one of the most convenient forms of apparatus to operate is the double effect with the single effect to get the final concentration in order to keep the initial steam pressure as low as possible.

The corrosive or embrittling action of caustic soda on steel evaporator tubes is very peculiar in that the effect is largely local, appearing at the upper end of vertical tubes just below the point where they are expanded into the flue sheet. No satisfactory explanation is yet put forward to account for this. An article on the subject appears in the March number of the Transactions

of the Faraday Society. Steel having undergone a somewhat similar caustic treatment has been shown to contain hydrogen.

E. M. SERGEANT: I am very much interested in this paper, particularly in the conclusion which he draws in regard to the relative disadvantage of the triple effect, because that conclusion has been forced on me by experience; and I am glad to see it confirmed by such good authority.

I would like to know on what Mr. Mantius' figures for steam consumption are based. I notice in evaporating in triple effect 20,000 lb. of soda, that there was a consumption of practically 100,000 lb. of steam per day, or about 5 lb. of steam per pound of soda. I think that is considerably better than can be obtained in actual practice right along, considering all the things that happen—leakage and other things. According to my experience the actual consumption that you get, if you figure the coal that goes under your boilers, per pound of caustic you get out, will be a great deal more than that.

The tube question, of course, is very important. It is a very serious question and the article in the Transactions of the Faraday Society, to which Mr. Vorce referred, is a very complete summary of results, and in my judgment is exactly in line with what happens.

It would be very interesting if any one could give any practical experience in regard to the use of tubes containing a rather higher amount of carbon than usual, as suggested in the experiment referred to in that article. I have started some experiments with such tubes, but they have not gone far enough to give any results. If any one could give any actual results on that subject it would be exceedingly interesting.

OTTO MANTIUS: The corrosion of steel or charcoal iron tubes in evaporators handling electrolytic caustic liquors above 40° Bè is a source of considerable trouble and constant expense to all manufacturers. Aside from the question of poor quality of the material and bad workmanship in expanding the tubes in the flueplates, I believe that the following three factors determine the life of the tubes:

First. A chemical action by the highly concentrated caustic, as explained by Mr. A. H. Hooker.

Second. A mechanical or abrasive action by the salt passing along the surface of the tube with considerable speed. It is very natural that this abrasive action will increase with the amount of salt and the speed at which it passes through the tube. While it is true that in a double or triple effect the actual amount of salt to be separated is always larger in the first effect or first and second effects than in the last effect, we must not forget that on account of the higher specific gravity and the more rapid circulation the liquor in the last effect will always carry the largest amount of salt in suspension. For instance, in a triple effect the speed in the tubes of the last pan will be about eight times the speed in the first evaporator.

Experience has shown that the tubes after a certain period of service have still retained the original thickness near the lower flueplate, but have worn off and become thinner the nearer we come to the top. Aside from the chemical action this is largely due to the increased speed at which the salt passes through the tube. Speeds of from 30 to 40 feet per second in the upper part of the tube are quite common in evaporators working at from 27 to 28 inches vacuum. As no vapor is developed in the lowest part of the tube the speed will not be more than 3 or 4 feet under the same conditions.

Third. A physical action which tends to make the iron very brittle near the top and causes circular cracks. So far very little is known on this subject. The Imperial Physical Testing Laboratory in Germany made a series of tests more than ten years ago without giving any tangible results. The article in the March number of the Transactions of the Faraday Society seems to give the best explanation.

The amount of salt given as 100 grams per liter is probably somewhat low for the average liquor, but I have been told that some plants are working with such a percentage of salt. An increase to 180 or 200 grams per liter will not change the relative value of my figures, as it will increase the steam consumption in all cases.

The data for steam consumption were taken from actual tests made with a plant handling 10 tons caustic per day, by weighing the condensation coming from the first effect. Such results might

not always be obtained in a yearly average, but I have seen records of a plant handling about 30 tons caustic per day in a triple and single effect, which record shows that only 4 lb. of steam are used for each pound of caustic.

Regarding the steam pressure used for the operation of caustic evaporators, it makes practically no difference so far as steam consumption is concerned, whether the first effect received the exhaust at 7 lb., 5 lb. or 0 lb. pressure.

The combination of a double effect and single effect is very good for small capacity, but the steam saving in a triple effect and single effect for larger plants is considerable and the operation just as simple.

SYMPOSIUM ON THE PRACTICAL SIDE OF ELECTROCHEMICAL INVESTIGATION

F. A. LIDBURY: This will be an Experimental Symposium in two senses. It is to consist of a number of experimental demonstrations of apparatus and methods such as are used in commercial and industrial electrochemical laboratories in the course of electrochemical investigations, as well as in other chemical investigations best or most conveniently carried out by electrochemical methods. It is true that to a great extent the methods and apparatus will be familiar to most of you from previous publications or descriptions. But actual demonstrations have the advantages of giving much clearer conceptions of the nature of such methods, of conveying a more exact and intimate knowledge of the details of procedure and of suggesting more clearly the scope. Discussion of the demonstrations will also permit a clearing up of points which in printed descriptions are sometimes overlooked. The point which it has been the intention of the organizers of this Symposium to emphasize is one which, while it holds fairly generally in all branches of the technical application of science, happens to be particularly easily and fully illustrated in electrothermic lines. This point is, that in commercial investigation the elaboration of method and apparatus, which generally characterizes academic research, gives way to simplicity. The reason for this, of course, is to be found, not mainly in consideration of expense, but in consideration of time. In industrial and commercial work the time element is not infrequently the most important. He will "get there" most quickly who is the most skilled in the construction, from simple and ordinary materials which lie always at his hand, of the simplest and sturdiest means of enabling him to make what tests or measurements he wishes. Under such circumstances, the excision of unnecessary elaboration results not only in the vital economy of time, but also in incidental financial economy without

any loss of essential accuracy. Indeed, true accuracy consists in the proportioning of the means to the end; and he cannot be said to possess it, who, in analysis, weighs to 0.01 percent when he requires a result no closer than 1 percent. This Symposium is therefore intended primarily to demonstrate, though necessarily in a very incomplete manner, how the simplest possible "home made" electrothermic apparatus is applied to the kind of investigation in which many of us are engaged.

In a second sense, this Symposium will be experimental in its order of procedure. It is desired to combine the demonstrations of the various apparatus placed to your left with discussions which, for the sake of order as well as to obtain a transcript for the Transactions, must necessarily be of a semi-formal nature. We will therefore adopt the following experimental procedure, subject to change if it is found not to work satisfactorily. The demonstrator will in each case ascend the platform and shortly explain what he is going to show. The demonstration will follow and you are requested to pass the apparatus in file. When everyone has viewed the demonstration the meeting will again be called to order, and discussion, which should be limited as much as possible to question and answer, will follow.

A SIMPLE LABORATORY TRANSFORMER SET.

F. A. J. FITZGERALD: This is a demonstration of an apparatus which is found very useful for many small electric furnace experiments. The diagram shows the method of connecting the four transformers, 100-volt primary and 10-volt secondary. By various combinations a considerable variation in voltage is obtained as shown by the following table of connections:

Switch Combination	Secondary Volts	Maximum Current	Maximum Kw.
1-3-5-8-10-12	40	125	5
1-3-5-8-9-12	20	250	5
1-3-5-7-9-11	10	500	5
2-3-6-7-9-11	5	500	2.5
2-4-6-7-9-11	2.5	500	1.25

By means of the rheostat R the voltage of the primary circuit can be regulated so as to vary the voltage between steps in the secondary circuit.

In order to illustrate the working of this apparatus a graphite rod 6.5 mm. (0.25 inch) in diameter has been mounted between carbon terminals placed 200 mm. (8 inches) apart. It will be seen that we can accurately adjust the temperature of this rod as desired (Fig. 2). Finally, when it is surrounded with sand, the secondary e. m. f. adjusted to 20 volts and a current of 200

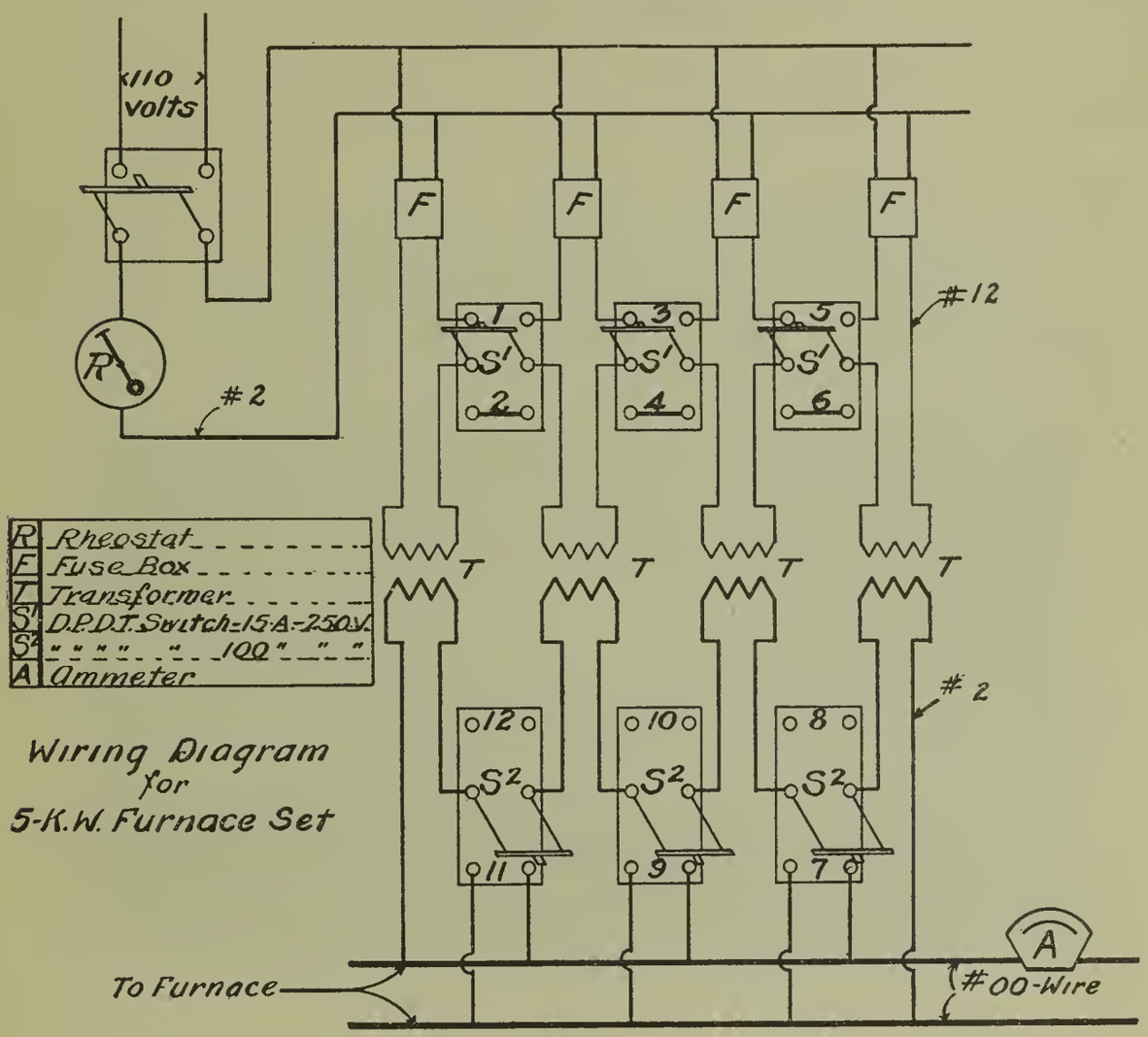

FIG. 1.

amperes obtained, a very fair fused silica tube is formed in about 6 or 7 minutes. This method of making silica tubes was first described by Despretz.

W. R. WHITNEY: How do you fasten the carbon rod in the furnace terminals?

F. A. J. FITZGERALD: Simply by making slots in the terminals into which the ends of the graphite rod fit very loosely and then making good contact by packing in graphite powder.

W. R. WHITNEY: Can you purchase those small transformers

without the casings? Is there any economy in getting them as you have them on the wall?

F. A. J. FITZGERALD: These transformers were made by the Pittsburgh Transformer Co., and I suppose are cheaper in that form. The whole outfit did not cost much more than $100.

MUFFLE FURNACE.

L. E. SAUNDERS: A furnace for the production of high temperatures in a small space can be constructed in an inexpensive manner according to the experiment shown. Its essential parts are:

1. A carbon or graphite resistor, made by sawing a block, plate or tube, in such a way that the path of the current is greatly lengthened.

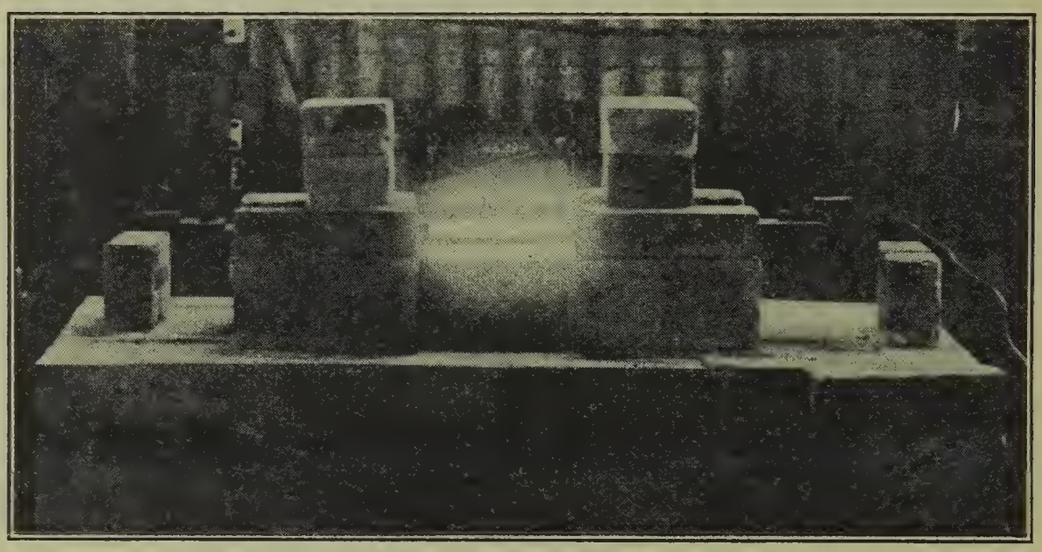

FIG. 2.

2. A pair of carbon heads cut out, so that the ends of the resistor element will fit loosely.

3. Brick-work sides, bottom and top all protected with a thick (2-inch) layer of firesand moistened with dextrine or other temporary binder.

4. An entrance or peek-hole door made from a piece of carbon with sight holes closed by removable plugs.

5. Copper contact plates bolted to the carbon terminals, or cables directly attached to the carbons.

In the experiment shown a granular carbon resistor has been placed in the circuit so that it may be conducted in the usual

lighting circuit voltage, 110. If a voltage as low as 20 to 30 is available no granular resistor is needed, and the desired temperature may be decided upon after a few experiments by changes in the resistor, such as thickness, length, distance between saw cuts, etc. The Acheson graphite resistor plate shown here is 12 inches (31 cm.) long, 4 inches (10 cm.) wide and one-half inch (13 mm.) thick and has 20 sawcuts made alternately on opposite sides, making the actual resistor a zigzag square rod about one-half inch (13 mm.) square and 40 inches (102 cm.) long,

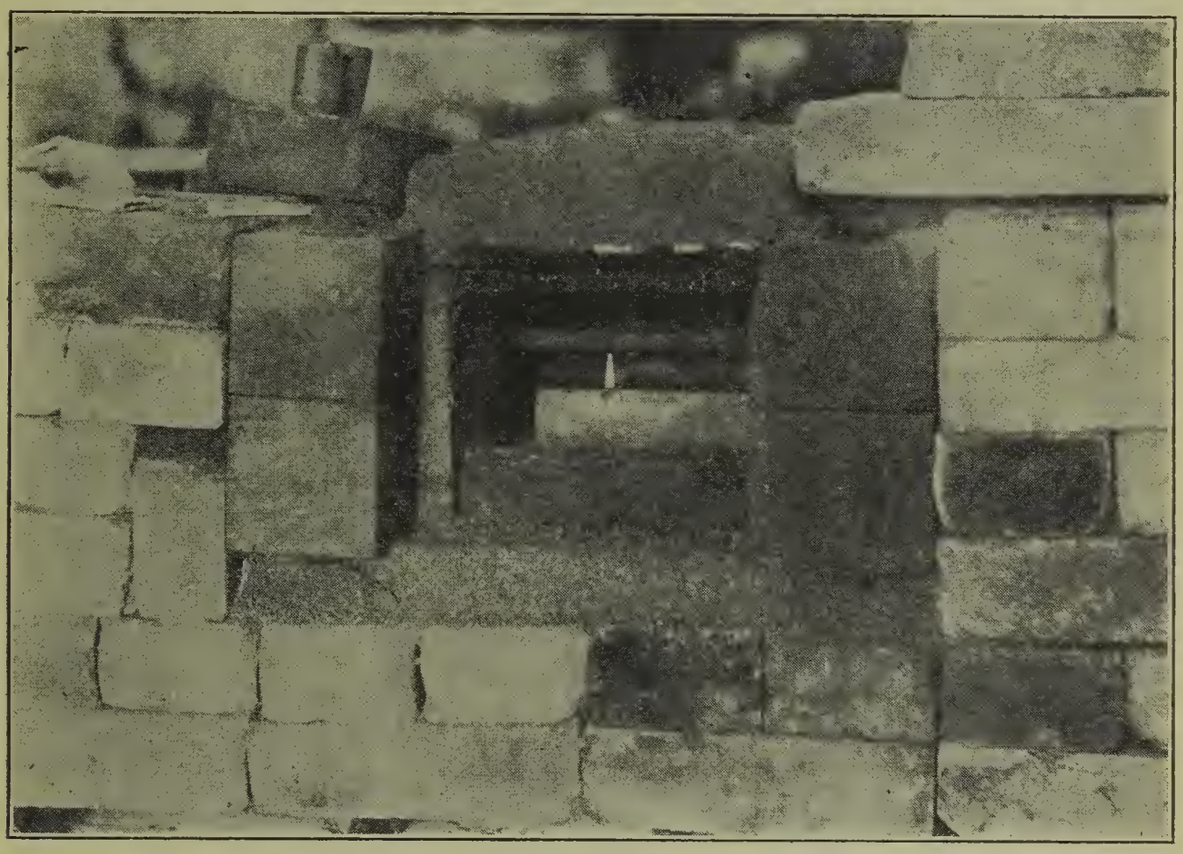

FIG. 3.

except the turns where the cross-section increases slightly. The plate is allowed play at the ends for expansion when hot, but is packed firmly into the carbon terminals with powdered graphite. To produce a temperature of 2,000° to 2,500° C. requires a power consumption of from 12 to 20 kilowatts, depending on the heat insulation installed and the accuracy of the sawing of the carbon plate. One plate will last for five or six experiments of moderate length, after which heating is likely to be uneven, due to oxidation of the resistor.

At temperatures no higher than 1,700° C., carbon resistors may be used. Beyond that, graphite is preferable, as there is a

rapid disintegration of carbon articles under these conditions at these temperatures, not noticeable with graphite.

In the furnace as shown, articles to be heated are placed under the resistor, the heat being reflected downward, and obviously the points closest to the graphite grid are hottest. This makes it extremely useful for melting-point work, as samples made up into cone shapes are melted at the tips where sighting with a pyrometer is easy.

Numerous modifications of this furnace will suggest themselves to any user of even moderate ingenuity.

A rather full description of some other forms has been published by Mr. F. A. J. FitzGerald in Vol. III, Electrochemical and Metallurgical Industry, 1905.

The total cost of this furnace should not exceed twenty-five ($25.00) dollars. (See Fig. 3).

TUBE FURNACE.

L. E. SAUNDERS: The second furnace which I shall show is a simple carbon tube, through which an electric current is passed by means of carefully fitted graphite blocks at the ends and these in turn connected by cables to an alternating current circuit. This length, five feet (152.5 cm.) has been chosen in view of the transformer conditions prevailing. The lowest voltage from the transformer is 50, and by previous experiment it has been found that five feet of carbon tube, 1½ inches inside diameter by 2 inches outside diameter (3.8 x 5.1 cm.) will heat rapidly to 2,000° C. at this voltage, drawing a maximum of 1,600 amperes.

The precautions to be taken are mostly in the contacts. These here are six inches (15.2 cm.) long made by hand from graphite blocks and the hole is slightly larger than the outside diameter of the tube, the remaining space being filled tightly with soft powdered graphite.

The treatment is very severe, for the temperature increment is very rapid, as may be seen. Tubes of careful construction are needed, the one in use being a stock article from the National Carbon Company.

It is only necessary to have more voltage variations available, to change the length and size of tubes used. The important point is the knowledge that tubes may be obtained to withstand such misuse.

The furnace as shown is very convenient for melting-point work where measurement of temperatures with optical instruments is desired. Fume troubles may be avoided by the passage through the tube of a neutral gas such as hydrogen. If "black body" conditions are not present on account of shading off in temperature near the ends, a disc of carbon may be shoved in ahead of the specimen and used as a ground for sighting against.

In the present case a temperature of 2,000° C. may be reached in about 15 minutes, and more rapidly if a brick or other refractory wall surrounds the tube, leaving a slight air space, preferably. This will of course prolong the life of the tube, which has been frequently used for ten to fifteen experiments under these conditions.

The total cost of this furnace without cables will not exceed fifteen ($15.00) dollars.

WIRE-WOUND ELECTRIC FURNACES.

J. B. Glaze: I have not come prepared to give a talk on all conditions and sizes of laboratory electric furnaces, but I intend to give a demonstration on the winding of a small crucible furnace. In the Norton laboratory no gas is used, so that electric furnaces take the place of all operations and heating formerly done by gas. This condition has enabled us to construct very cheaply the various types and sizes of furnaces now in use in our laboratory. It is not only the cheapness of the materials, but also the speed with which you can put one of these furnaces into operation that prompted this demonstration and exhibit.

I brought two types of furnaces for you to see in operation: one is a tube furnace, the other is a small crucible furnace. With the proper materials at hand, I expect to show how quickly a furnace can be wound, covered with alundum cement, packed in the iron shell with Kieselguhr, connected directly across a 110 volt circuit and brought to about 1,000° C. temperature. The materials to be used are:

Alundum Furnace Core, 2-1/16 inches bore by 2-7/16 inches depth (5.2 x 7.5 cm.).

Iron Shell Container, 6 inches in diameter by 5 inches depth (15.2 x 12.7 cm.).

Kieselguhr for insulation.

Nichrome Wire No. 20 (B & S) 38 feet (11.6 m.) in spiral coil.
Alundum cement for covering wire on core.
Alundum cover for furnace core.
Asbestos cover for iron shells.

The iron shell container may be made out of sheet iron or any other material that will not be affected by heat. Legs may be attached to this to hold it off the table or bench. The furnace core may be larger or smaller according to the requirements of the furnace. For this size of core (2-1/16 inches bore by 2-7/16 inches depth) a temperature of 950° C. may be obtained by using 38 feet of No. 20 Nichrome wire on 110 volts with a power input of 400 watts. The core and wire must be carefully covered with cement to protect the wire from oxidation and any impurities in the Kieselguhr that may unite with the wire at 950° C. The cement prolongs the life of the wire. The temperature, of course, depends greatly upon the material used for insulating the core, but with Kieselguhr we have obtained good results and by using this insulating material in all our furnaces the temperature varies with the power input.

If the temperature goes over 1,050° C. the Nichrome wire oxidizes very rapidly. This was also noticed in the tube furnace you see on the bench. This tube furnace reaches a temperature not over 850° C., but the draft through the tube was so strong that the wire was oxidized through the porous tube at that temperature. The latter difficulty was avoided by using the glazed tube, which prevented any oxidation. The glazed tube shown with the wire still in good condition was in service seven months and is good for much more use. The power input for the tube furnace is 600 watts. The tube is 1¾ inches bore with walls ¼ inch thick and 16 inches (4.4 x 0.6 x 40.6 cm.) long. Only 12 inches (30.5 cm.) are enclosed in the iron case. Both ends of the tube are open.

The power input for any such furnace depends upon the temperature desired and the size of the furnace, or square inches of surface to be heated. Some idea may be obtained from the figures I have given. The cost of these furnaces is very low.

I will give a demonstration of the winding and assembling of the crucible furnace. The furnace will be started, and with a

wattmeter connected in the circuit you may see the power input for each furnace.

Jos. W. Richards: Has Mr. Glaze used any other wire than the Nichrome wire, such as molybdenum, or others, for higher temperatures? How high a temperature will a furnace stand?

J. B. Glaze: The furnace will stand, of course, as high a temperature as the Nichrome wire. We have not used anything else but the Nichrome wire. The wire will stand about 1,050° C. The oxidation is quite rapid at that temperature. I think the No. 2 Nichrome wire is guaranteed for 1,100°, but the furnaces we have had ranged between 900 and 1,000° C. That is as high as we desire in our work, so we have not tried to go any higher. The crucibles themselves will stand a much higher temperature.

ELECTRODE JOINTS.

A. T. Hinckley: The subject of my talk is, "The Electrical Resistance of Electrode Joints." This method of measuring the electrical resistance of electrode joints is the same as described by FitzGerald and Hinckley before this Society in 1913 (Trans. Amer. Electrochemical Soc., **23**, 333), and consists in comparing the potential drop in a given length of electrode including a joint, with the potential drop in a similar length without a joint.

In the test being shown, a 10 x 60 inch (25.4 x 152.5 cm.) jointed electrode is used with its lower end in a carbon resistance to take up the load. A current of 4,000 amperes, 50 amp. per square inch, is passed through the electrode and the potential drop measured at the joint. Using the symbols given in the article referred to

$$x = \frac{C\,E'}{E''} - (a + b)$$

$$= \frac{6 \text{ x } 2.4}{1} - (6 + 6)$$

$$= 2.4$$

This means that the resistance of the joint is equal to the resistance of 2.4 inches (6.1 cm.) of the solid electrode. From this we may calculate the percent of total energy lost in the joint.

Electrode resistance, 20° C. = 0.0018 ohms per inch cube.
Resistance of 1 inch of length = 0.000023 ohm.
Resistance of joint = 2.4 x 0.000023 = 0.000055 ohm.

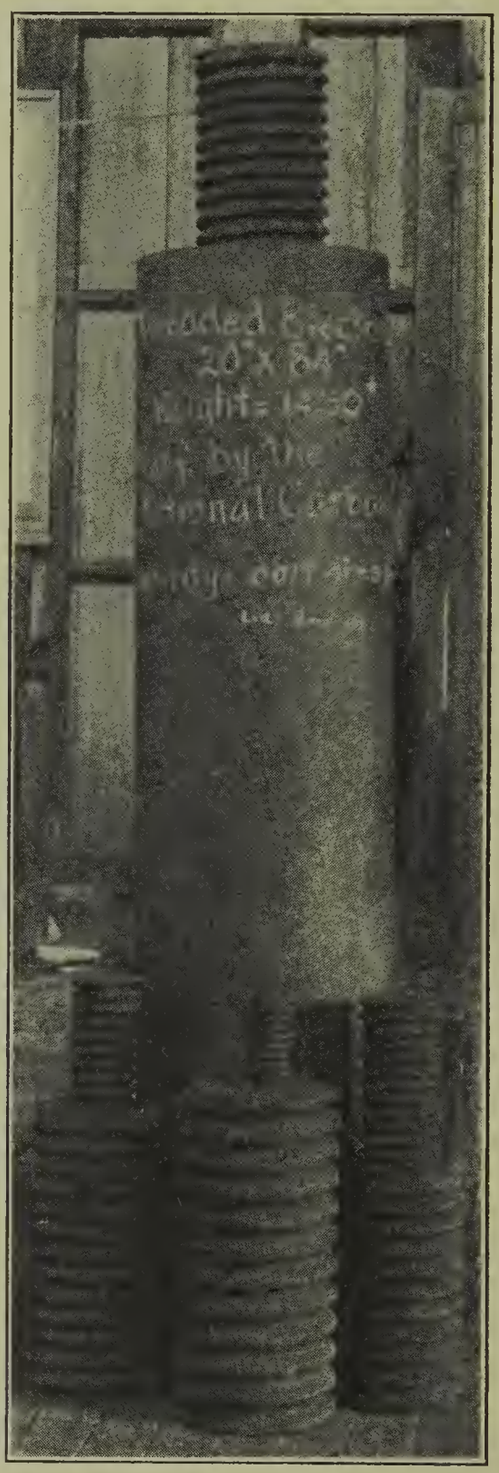

FIG. 4.

Energy lost in joint = I^2R = 4000^2 x 0.000055 x 0.001 = 0.88 kilowatt.

Total energy = 200 kilowatt.

Percent energy lost in joint = 0.88/200 = 0.44 percent.

This means that the energy being lost at the joint is only 0.44 percent of the total energy being developed in the furnace.

Jos. W. Richards: Has Mr. Hinckley had occasion to drill these carbon electrodes, and if so has he any objection to telling us what is the best material to use for drilling the holes in the carbon electrodes?

A. T. Hinckley: I think the Rex A tool steel stands up as well as anything.

Jos. W. Richards: What sort of paste makes the best composition for joining electrodes?

A. T. Hinckley: I would recommend some paste without a carbonaceous bond, such as the Electrode Joint Compound made by the National Carbon Co. (See Fig. 4).

REFRACTORIES.

A. T. Hinckley: Many cases arise in furnace work where it would be desirable to use carbon as a refractory material if means could be found for decreasing its rate of oxidation and decreasing its electrical conductivity. The "Vitricarbo" refractories were developed for this purpose and, therefore, contain a vitrifying ash.

Two grades of "Vitricarbo" are manufactured at present.

Grade B-10 is a coarse-grained material, made to withstand rapid thermal changes without cracking. It has been tested up to about 2,000° C. under neutral or reducing conditions without softening or material shrinkage. The apparent density, 20/4°, is about 1.70 and the porosity about 23 percent. The heat conductivity is less than carborundum brick and greater than alundum brick, as measured by the method described by Fitz-Gerald, Trans. Amer. Electrochemical Soc., **21**, 535. The electrical conductivity is about one ohm per inch cube at 20° C. and the crushing strength about 500 lb. per square inch.

Grade B-11 is finer grained than B-10 and is used where greater crushing strength is needed (1100 lb. per square inch). Its properties are similar to B-10 except that its electrical conductivity is slightly greater.

These brick show very little corrosion from vapors of iron or lime in spite of their acid constituents, as will be shown in Mr. FitzGerald's experimental arc furnace for melting lime.

When "Vitricarbo" brick are used in a furnace roof, a covering of heat insulation brick should be used on the outside to protect the "Vitricarbo" from oxidation and to cut down the heat losses. "Vitricarbo" brick should never be used under strongly oxidizing conditions.

These samples (exhibiting brick) show the relative amount of dripping from a silica brick, a high-grade fire brick and a "Vitricarbo" brick placed 4 inches (10 cm.) above a granular carbon resistor, running at a temperature of about 2,000° C. The heat was maintained for 20 minutes, and you see that the "Vitricarbo" brick dripped less than either of the others. The atmosphere was of course reducing.

Another experiment was made cutting little pyramids from "Vitricarbo" brick and silica brick, which were placed on a carbon slab with Seger cones up to No. 39, which melts above 1,900° C. These cones were heated gradually in a carbon tube furnace and the "Vitricarbo" (exhibiting sample) showed no softening at a temperature well above that at which cone No. 39 melts. When the temperature is raised still higher the vitrifying ash is vaporized leaving graphite. This is shown by this brick (exhibiting sample) one end of which is partly converted into amorphous silicon carbide and the other end into graphite.

In studying heat insulation brick, we have found that practically none of the grades now on the market will stand direct exposure to heat above about 1,000° C. The grade HI-10 was, therefore, developed to answer the demand for a brick with properties intermediate between the ordinary high grade fire brick and heat insulation brick of the kieselguhr type. This brick can be heated to 1,400° C. without softening and there is very slight shrinkage on repeated heating and cooling. The heat conductivity is about 70 percent as great as that of a high-grade fire brick at 700° C. Porosity is about 60 percent and apparent density about 1.10 at 20/4°. Crushing strength about 500 lb. per square inch.

Jos. W. Richards: I would like to ask two questions. One is, approximately the commercial price of this "Vitricarbo" brick;

the other is, whether the absence of corrosion is due to its very high melting point or to its great conductivity for heat.

A. T. HINCKLEY: The price is between that of silicon carbide brick and silica brick.

We believe that the absence of corrosion is due to the presence of the carbon particles.

JOS. W. RICHARDS: Does the electrical resistance decrease with the heat?

A. T. HINCKLEY: Yes.

ALFRED H. COWLES: What is its composition?

A. T. HINCKLEY: It is an acid refractory.

RADIATION ARC FURNACE.

F. A. J. FITZGERALD: The principle of this furnace is due to Siemens[1] and is used by Stassano in his well known furnace. The purpose of the demonstration is to show how cheaply a furnace of this kind can be made with pipe fittings (See Fig. 5).

We shall show how a quantity of lime can be fused in this furnace in 7 or 8 minutes, using a current of 1,200 amperes at 50 volts.

The cover of the furnace is made of what is known as "Vitri-carbo" brick, which has been described by Mr. Hinckley.

MACHINING ACHESON-GRAPHITE.

EDWARD R. COLE exhibited the machining of Acheson-Graphite articles of various kinds (Fig. 6). He said in part:

This tube was machined from a solid Acheson-Graphite electrode, for use as a resistor for an electric furnace. You can see that it is very resilient and will stand severe mechanical strain without breaking. In fact we have cut a 2-inch (5 cm.) diameter spiral with 16 turns per inch (6 per cm.) having a total length of 12 inches (30 cm.) which was bent until both ends met and was elongated 6 inches (15 cm.) without injury.

Among the most unusual uses is this one of substituting graphite for the rubber Fuller Ball of a compression cock. These were first made for the Bureau of Standards at Washington.

[1] British Patent 4,208 (1878).

Another article attracting considerable interest is a fluted one. Several have asked if it is a milling cutter, as it very much resembles one in appearance. It is a terminal for a mercury arc rectifier, and may be seen in operation at moving picture houses,

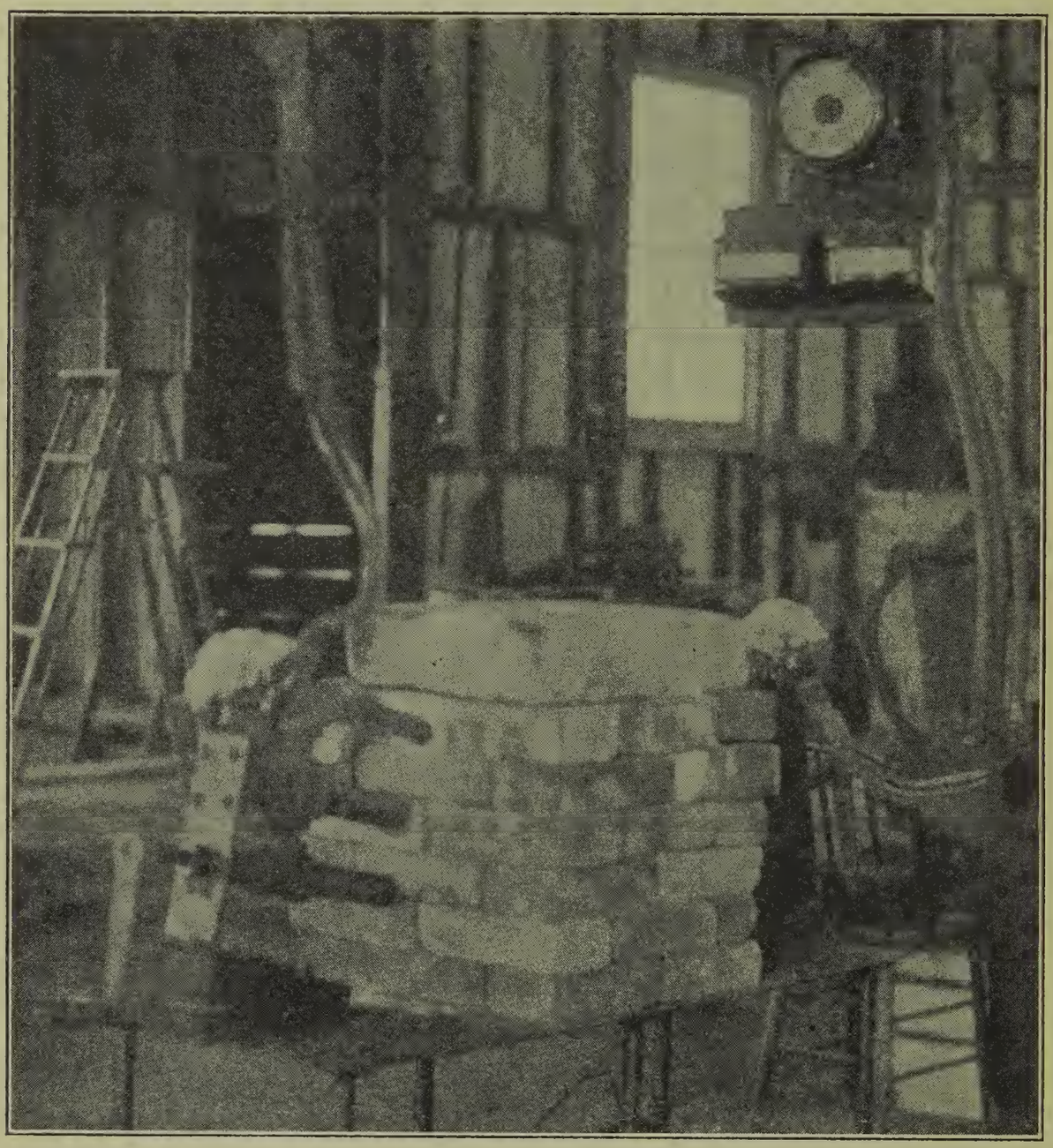

FIG. 5.

where the rectifier is placed in a conspicuous place to attract attention, the two black objects to which the terminals of the tube are attached being these graphite electrodes.

This tube is made with the greatest accuracy possible. It must be within 0.005 inch (0.127 mm.) of given dimensions, and must

have a very homogeneous structure. These tubes are used in the study of the spectrum.

Precision and accuracy, however, enter into the machining of a great many other articles. Some of the discs are machined within 0.002 inch (0.05 mm.) of a given thickness and must be perfectly flat. Thirty-two of these when piled one upon the other must not show any light between points of contact, nor vary 0.03 inch (0.76 mm.) above or below a total height of 2 inches (5 cm.).

This sample of accurate machining shows a method of pinning two pieces of graphite together with a tapered pin. After the pin was driven into the hole with a light hammer, the piece was sawed through to show the fit of the pin into the hole. Careful examination of this piece shows that if it were not for the differ-

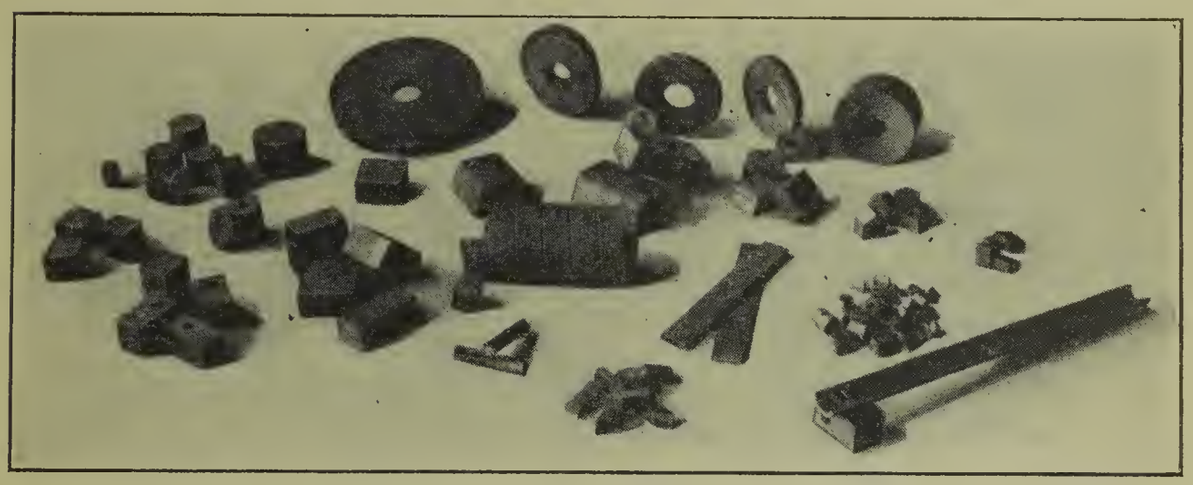

Fig. 6.

ent directions of the grains of the material, it would be impossible to find the pin. We recently completed an order on which over 5,000 of such pins and joints were used.

It is practical to use Acheson-Graphite electrodes to replace platinum points on delicate instruments and relays, the copper plating making it possible to solder the contacts into the holders. The specific resistance per inch cube of these contacts is 0.00032 ohm, or 0.000813 ohm per centimeter cube. (See Fig. 6).

MOLYBDENUM RESISTANCE FURNACE.

C. Dantsizen: Molybdenum and tungsten resistance furnaces are described in the Transactions of the American Electrochemical Society, **20**, 287 (1911). Since that date furnaces of this type

have been used extensively in our laboratories for making alloys, high temperature insulators, etc., and have become more or less standardized. A furnace of the form shown requires 60 volts and 60 amperes to maintain a temperature of about 1,700° C., and from 2 to 3 cubic feet (57 to 85 l.) of hydrogen per hour to protect its molybdenum from oxidation; 1,700° C. is reached in about an hour's running on constant current, the voltage being gradually raised as the temperature rises. In our laboratories this change in potential is generally obtained by varying the field current of a small 1,200 volt A. C. generator connected through a 10 to 1 transformer to each furnace. Other methods of changing the voltage are the use of resistance in series with the furnace, a voltage regulator, or a group of transformers such as Mr. FitzGerald showed.

In designing a furnace the following data are used:

1. Resistance of molybdenum at 0° C. is 4.1 microhms per centimeter cube.

2. Co-efficient of resistance of molybdenum is 0.005. This makes its resistance at 1,700° C. about 8.5 times its resistance at ordinary temperatures.

3. Six watts per square centimeter of heating surface should be allowed to maintain a furnace at 1,700° C.

4. There should not be more than one volt per 2.5 millimeters between turns in the molybdenum winding. For example, if two adjacent turns of a winding are 7.5 millimeters apart, a drop of more than 3 volts in a single turn of the winding might lead to a breakdown in the insulating alundum tube, especially at the higher temperatures, thereby giving rise to an arc which will burn out the winding at that point.

5. Molybdenum wire may be obtained in any size from 15 mils to 100 mils in diameter. (Sizes of the wire are given in mils; that is, thousandths of an inch.)

Most furnaces have to be built to fit some definite maximum voltage, such as 60, 110, 220, etc., and a calculation of length and diameter of wire is then necessary. Alundum tubes may be obtained threaded and in this case the length of the winding is easily determined.

The following example might be given for showing the calculation of the diameter of the wire for a small furnace. Suppose one can obtain a varying potential from 2 to 60 volts and wishes to build a furnace using an alundum tube 24 centimeters in circumference, 25 centimeters long, and threaded so that the distance between turns is 1.25 centimeters. About 5 meters of wire will be required, and, as there are 20 turns in the length of the tube, there will be for each turn 60 divided by 20, or 3 volts drop, which is well within the limit given in the data above. The area of the heating surface is 24 x 25, or 600 square centimeters, and to maintain this surface at 1,700° C., 600 x 6, or 3,600 watts, will be required. With 60 volts the current required will be 60 amperes and the resistance will be 1 ohm for the entire length of the winding, or 0.2 ohm per meter. From the following table one finds that wire about 60 mils in diameter should be used in the construction of this furnace.

Diameter in Mils.	Resistance per Meter of Molybdenum at 1700 °C.
15	3.04
21	1.54
23	1.41
25	1.095
30	0.762
40	0.428
50	0.274
60	0.190
70	0.1395
80	0.107
90	0.0847
100	0.0685

Powdered flint, alumina, and magnesia have been used as heat insulators for these furnaces.

Gases which have been employed to protect the windings from oxidation are hydrogen, nitrogen, coal gas, and methyl alcohol vapor. Unless very pure nitrogen can be obtained it is advisable to mix about 5 percent hydrogen with it before passing it into a molybdenum furnace.

In the making of alloys we have used crucibles pressed from pure alumina and large enough to hold 100 grams of molten iron.

HERING FURNACE.

CARL HERING: In a paper[4] read before this Society some time ago I described a type of electric furnace based on a new electromagnetic principle or force; as this force is not yet described in text books and may therefore not yet be well known, I have brought the apparatus here exhibited to show this peculiar force and the way it is applied as the basis of an electric furnace. The device, as shown here, constitutes a real furnace when properly mounted, and is of the kind to be used for heat treating, like tempering, annealing, heating drop forgings, etc., by immersion in heated lead, but it is here mounted so as to keep the bath as cool as possible, as the mechanical motion produced by this force can then be better inspected.

This new principle is as follows. When a current passes through a conductor the magnetism in and about this conductor and produced by the current, tends to crush it, as the surrounding flux acts like stretched rubber bands. This crushing force is not strong enough to be noticeable in solid conductors, but when they are liquid, and when the current is large enough, its action becomes very evident; it is then often great enough to completely sever the conductor, and as this generally occurs locally at a weak spot, the conductor acts as though it were being pinched, for which reason I gave it the name of the "pinch phenomenon"[5] and called this radial force the "pinching force," which names have since come into use.

If now the conductor consists of a column of a molten liquid confined in a hole in a refractory material near the bottom of a crucible, and if the outer end is sealed by the electrode and the inner end opens into the bath in the crucible, the resultant action of this radially crushing force will force the liquid out through the center of the open end, and as this end is submerged the conductor cannot pinch off, but instead liquid will be sucked in circumferentially, thus producing a simultaneous outward and inward flow of the liquid through the hole. This outward flow of the molten lead is shown quite strikingly in this apparatus. You will see that the force is quite great. The mouth of the hole

[4] A New Type of Electric Furnace, Trans. Amer. Electrochem Soc., Vol. XIX, 1911.

[5] A Practical Limitation of Resistance Furnaces; The "Pinch Phenomenon," Trans. Amer. Electrochem. Soc., Vol. XI, 1907. The Working Limit in Electrical Furnaces Due to the "Pinch Phenomenon," Trans. Amer. Electrochem. Soc., Vol. XV, 1909.

is here about an inch or two below the surface, and when the round bottom of a ladle is pressed against it as though to close it, the molten lead will be squirted up an inch or two above the surface.

Due to this very rapid circulation of the molten material, many kilowatts of heat can be set free in the conductors in relatively small holes, as this heat will be rapidly transmitted to the main bath by the flowing material and distributed in it. This is the principle of this type of furnace.

In the present apparatus the crucible is a steel tank filled with molten lead. In it there is floating a block of refractory material the top of which is above the surface of the liquid. This block has two vertical holes in it from the top to near the bottom; into these holes dip the vertical electrodes which form the secondary terminals of the nearby transformer and lead the current into the furnace. Horizontal cylindrical holes below the liquid level extend from the bottoms of these electrode holes to the outside of the block and open out into the bath. Being submerged, they are always full of liquid metal, and it is in these cylindrical liquid conductors that the heat is generated by the passage of the current from the electrodes, and in which this peculiar force produces the rapid circulation, as you will see. You will thus see that the furnace proper, that is the heating device, consists of two holes in a refractory block, and holes are simple and cheap.

Although generally there is only one heating hole for each electrode, yet in the present apparatus there are two at an angle, like the letter V with its point at the electrode, the two pairs thus resembling the letter W, for which reason we call this the W-type. Although the same inward and outward flow just described exists in each of the four holes, yet there is then superposed another new force tending to cause an additional inward flow in the two middle holes and an outward one in the two outer ones. Physicists contradict the existence of any such axial force produced by neighboring conductors on each other, as such a force is not known in text books; you will see very clearly, however, that this axial force actually exists and is very effective. This inward sucking in the inner holes is great enough to produce an audible snorting noise when the level of the lead is only slightly

above the top of the opening. Most of our furnaces, however, have only one hole per electrode.

C. E. SKINNER: I should like to ask Dr. Hering how the furnace is started, in the case of the higher melting point materials.

CARL HERING: In starting these furnaces from the cold state they must be primed with a small charge of liquid metal, enough to fill the holes and connect their mouths, so as to establish the path for the current. With some metals, like in the present lead furnace, the charge may be allowed to freeze, and the heating may then be started again by simply applying the current gradually until sufficient of it is liquefied. With metals that are brittle, or hot-short, or contract greatly, it is safer to empty the charge completely when shutting down, and to start up again from the cold with a small charge of liquid metal. We have started frozen brass furnaces with the current, but do not at present recommend it as regular practice. Ferro-silicon, which can be melted very nicely in this furnace, is too brittle to be allowed to freeze in the furnace.

JOS. W. RICHARDS: I ask Dr. Hering about how much hotter the metal gets in the tubes than the metal in the baths, in other words, what is the rise in temperature of the metal? Did you measure it or calculate it?

CARL HERING: The difference of temperature between the holes and the bath depends greatly on the viscosity of the metal, its specific heat, the rate of input of energy, the pinching force, the velocity of flow, the length and diameter of the holes, and perhaps other factors also. Hence is not subject to calculation, except crudely. Moreover it naturally will be different in different furnaces. Rough calculations indicate that it may be 20° to 30° C. and perhaps sometimes as much as 100°.

JOS. W. RICHARDS: That might be settled by putting a pyrometer tube near the opening and measuring the temperature of the issuing metal.

CARL HERING: We intend to do it in this way, using a second pyrometer to measure the temperature of the bath at the same moment. It would not do to insert a pyrometer tube down into the hole, for unless very small, it would disturb the conditions

in the holes and therefore measure the temperature under abnormal conditions.

JOS. W. RICHARDS: The rise in temperature is probably very small; that is, considering the great amount of circulation. The rise of temperature in the tubes is only a few degrees C. or of that order.

CARL HERING: It depends greatly on the design and proportioning of the furnace and is complicated on account of the large number of variable factors involved. It is one of the features which governs the proportioning of these heating holes. In one case we found that the end of the electrode had not melted even when it had been in direct contact with the root of the resistor, hence the temperature there could not have been high.

JOS. W. RICHARDS: This question has bothered a good many people who have been reading about Dr. Hering's furnace, and who imagine that the temperature in these tubes must be several hundred degrees higher than the temperature of the bath. I satisfied myself by a calculation on one of Dr. Hering's furnaces that it would be something like 40° higher than the mean temperature of the bath in the extreme case.

M. G. LLOYD: What is the material of the electrode in contact with the molten metal?

CARL HERING: The same metal as that which is to be melted in the furnace, so as not to contaminate the latter. If brass is to be melted, or other copper alloys, we make them of copper, because an alloying with some copper would not do any harm. In a silver furnace the electrode is made of that metal only up to the point where it might melt, and from that point on it is copper. In a steel furnace the main part of the electrode is made of copper and only the end which may melt is made of steel. Graphite electrodes may in some cases be used.

JOS. W. RICHARDS: Has Dr. Hering tried the use of anything except metal, that is, any liquids or fused electrolytes, for instance, which are good conductors?

CARL HERING: It depends on their resistivity; when this is too high the proportions of the heating holes become impracticable,

although there are then other means which may be resorted to which I am not ready to describe now. The resistivity of barium chloride is too high, but we heat such materials by floating them on a bath of lead or other suitable metal in the bottom and setting free the heat in that metal. Similarly a laboratory furnace for heating any materials in a crucible may be made, in which case the crucible is forced down into the heated bath of molten metal; and by using a metal of sufficiently high boiling point, such a furnace might perhaps be made for a range of temperatures above that at which metal resistors melt or at which a carbon resistor disintegrates too rapidly.

Jos. W. Richards: May I ask how the brass furnace which you put up at Philadelphia is working? I know many here will be interested to know on what scale it is running and with what success.

Carl Hering: It has been working very satisfactorily as far as the furnace itself is concerned; many tons of brass have been melted in it; any desired temperature can be reached. Preferably a large part of the charge is always retained, as the cold metal then melts quicker by immersion, and the full current may then be kept on during pouring. It holds about 1,600 pounds. The power input is about 100 K. W., though this depends somewhat on the resistivity of the particular alloy and is in this particular furnace also limited by the range of regulation of the particular and rather poor transformers. The melting rate was found to be about 8 pounds per K. W. hour from switchboard to clean ingots cast, the raw material being a rather poor scrap. This includes some avoidable heat losses in the transformers and elsewhere, as also in the dirt in the scrap. With clean metal, better transformers and a reduction in some other losses, 10 pounds per K. W. hour will no doubt be obtained, and perhaps exceeded; but this depends greatly on the superheat desired by the foundryman. Our delays and troubles have been due mostly to things outside the furnace proper, and were largely in the transformers. Much delay was caused by finding that the counter electromotive force developed by the moving metal was far greater than expected; the voltage of the transformers was therefore far too low. After doubling it and halving the current, the furnace ran

very well. The counter e. m. f. seemed to be even greater than the voltage required by the resistance alone. This, of course, is a very favorable feature.

Jos. W. Richards: On the spur of the moment, I will confess I do not know what the counter electromotive force will mean in such a furnace.

Carl Hering: All the energy of the mechanical motion of the liquid appears in the circuit as a counter e. m. f. or opposing e. m. f., just as in an electric motor. It is similar to the counter e. m. f. in an electrolytic cell, in which case however it represents the chemical energy. The energy input therefore is made up of two parts, one is that which is converted directly into heat due to the resistance, and the other that which is converted first into the mechanical energy of the moving metal, and afterwards fritted into heat by friction. This counter e. m. f. is produced while the metal flows from the circumference of the hole to the center, during which time it cuts the magnetic flux of its own current, which induces a voltage like in a dynamo.

F. A. Lidbury: About what energy do you use in practice?

Carl Hering: The rates from actual runs on brass I have given above. But neither these nor those for other metals have as yet been determined with care. As there are only two losses in this furnace, that through the walls and that through the electrodes, there is no reason to believe that both may not be made small by better proportioning, hence the heat efficiency be made large. It has taken about thirty-five years to develop the best proportions of the arc furnace, and about twenty-five for the induction furnace; to determine the best proportions of furnaces is a slow process.

The question of the wearing of the walls of the holes has often been asked, the general opinion being that it would be serious; I feared it myself. It turned out however to be quite the contrary, to our surprise. For steel furnaces we use a refractory material made here in Niagara Falls, an electrically sintered magnesite, which gets very hard and on which we have not been able to detect any measurable wear. In one case a thin fin of this material was accidentally formed at the mouth of a hole during

the tamping of the plastic material. To my surprise it remained there for a number of runs, although tons of molten steel must have washed over it. Something in the steel seems to deposit on the surface of the holes, forming a black glaze which seems to replace itself as fast as it is worn away. In brass furnaces the holes are not thus wetted, and remain hard and smooth. For brass we find tubes of the ordinary crucible plumbago to be very satisfactory. In the preferred form of furnace in which the holes enter from the side, that part of the lining which includes the holes is easily and cheaply replaced; the plastic material is rammed around cores made of thin metal tubing, which latter are melted out when the furnace is started.

WARREN F. BLEECKER: Could this furnace be used as a reduction furnace?

CARL HERING: This type of furnace is primarily for developing heat in molten metals; whether this heat can subsequently be used for chemical reductions depends upon the process involved. Iron oxide was easily reduced to iron with the carbon in the iron, like in the open-hearth process, and more carbon can easily be dissolved in the iron; a rod of electric light carbon held in front of one of the holes for a little while comes out pointed, showing its rapid solution. Carbon floating on the surface no doubt reduces some of the floating oxides, as usual. Zinc, phosphorus, etc., could no doubt be reduced by mixing the raw material with carbon and floating it on some inert liquid metal in which the necessary heat is generated.

WARREN F. BLEECKER: The particular question arises as to whether the slag would interfere in any way, if you have some slag materials, and whether that slag will wash down in the metal and interfere with the furnace?

CARL HERING: Whether slag on top interferes with reduction depends on the particular process. Slag should not, and under normal conditions does not, get into the heating holes. Any small amounts of fusible slag suspended in the metal would soon be, and in fact has been, washed out of the holes if it got in.

WARREN F. BLEECKER: Would it be apt to do that?

CARL HERING: The only danger of slag in the holes is when the furnace is being emptied for a shut-down. With ordinary care this can be, and is, easily avoided by emptying the last dregs reasonably quickly.

F. A. LIDBURY: I do not know whether I apprehended what you said, but I gather that these troubles you had were chiefly in connection with the transformers. What was the nature of the troubles you met with? I realize you are using a transformer of an unusually low voltage, and it is not quite clear why you should have met with any particular troubles in that respect.

CARL HERING: It is true that many of our difficulties have been with the transformers, but although they were very time-robbing they were not inherent and could easily be avoided by better proportioned transformers. One difficulty was that the range of voltage was not great enough for the developing stage when it was not yet known just what voltage would be required. In several cases we found that the voltage had to be doubled, due to the unexpectedly high counter e. m. f., hence the transformers had to be reconstructed. Another was that the particular means for the regulation of the voltage in the transformer introduced a bad power factor into the transformer; this could be avoided by regulating it differently, as the power factor of the furnace is not low; at 25 cycles it was practically 100 percent, and at 60 cycles between 70 and 90 percent. Another was that the transformers were not designed liberally enough and therefore heated badly. Another was in the particular way in which the terminals had been made by the maker. All of our transformer troubles were of the kind that could be remedied by the transformer maker.

F. A. LIDBURY: What is the relation of the power factor to the capacity of the furnace as you have built it?

CARL HERING: There is no such relation; the power factor does not depend on the capacity. It depends more on the frequency and on the resistivity of the metal. It is high for low frequencies; it is lower for lower resistivities on account of the greater currents required. A low power factor merely means larger transformers and leads; it does not mean a lower efficiency

except indirectly, in that the losses are increased due to the larger idle currents.

F. A. LIDBURY: I presume the larger the furnace the lower the power factor is?

CARL HERING: No, not necessarily. A larger capacity furnace may be made by multiplying the heating units instead of the current in each; the power factor would then remain the same.

JOS. W. RICHARDS: Does Dr. Hering like to hear his furnace spoken of as a "pinch-effect" furnace? The pinch effect refers to something which does not happen in this furnace. You are using the pinch force, but do not get the pinch effect.

CARL HERING: Personally I do not care what you call it, as long as you do not call it bad names. The force of the pinch effect most assuredly is used, but we do not allow this force to rupture the circuit. But even when the latter does occur there is no harm done provided the metal is molten, for by then opening the circuit for a moment, contact is again established as the metal runs together again.

ELECTRO-PERCUSSIVE WELDING.

MR. CHUBB gave a demonstration of electro-percussive welding illustrating the paper by Mr. Skinner and himself on that subject given at an earlier session.

THE WILE FURNACE.

R. S. WILE: The furnace is a slag resistance furnace and is designed to use two- or three-phase current, while an older type in use for a number of years has one top and one bottom electrode and uses direct or single-phase current. (Fig. 7).

In starting this furnace the top electrodes are lowered and a quantity of granular graphite is thrown around the electrodes until arcing occurs; then broken glass is introduced. As this melts the electrodes are raised until the bosh is filled with it. The tin dross mixed with the proper amount of carbon is then placed in the furnace, completely filling the stack. As the metal is reduced it is tapped out at the bottom and provision is made to draw the accumulating slag through the slag tap hole at the

top of the bosh. It is necessary to add the proper amount of silica to the charge in case it contains iron, copper or other deleterious oxides in order to maintain the silica content in the slag constant, thus keeping the electrical resistance the same.

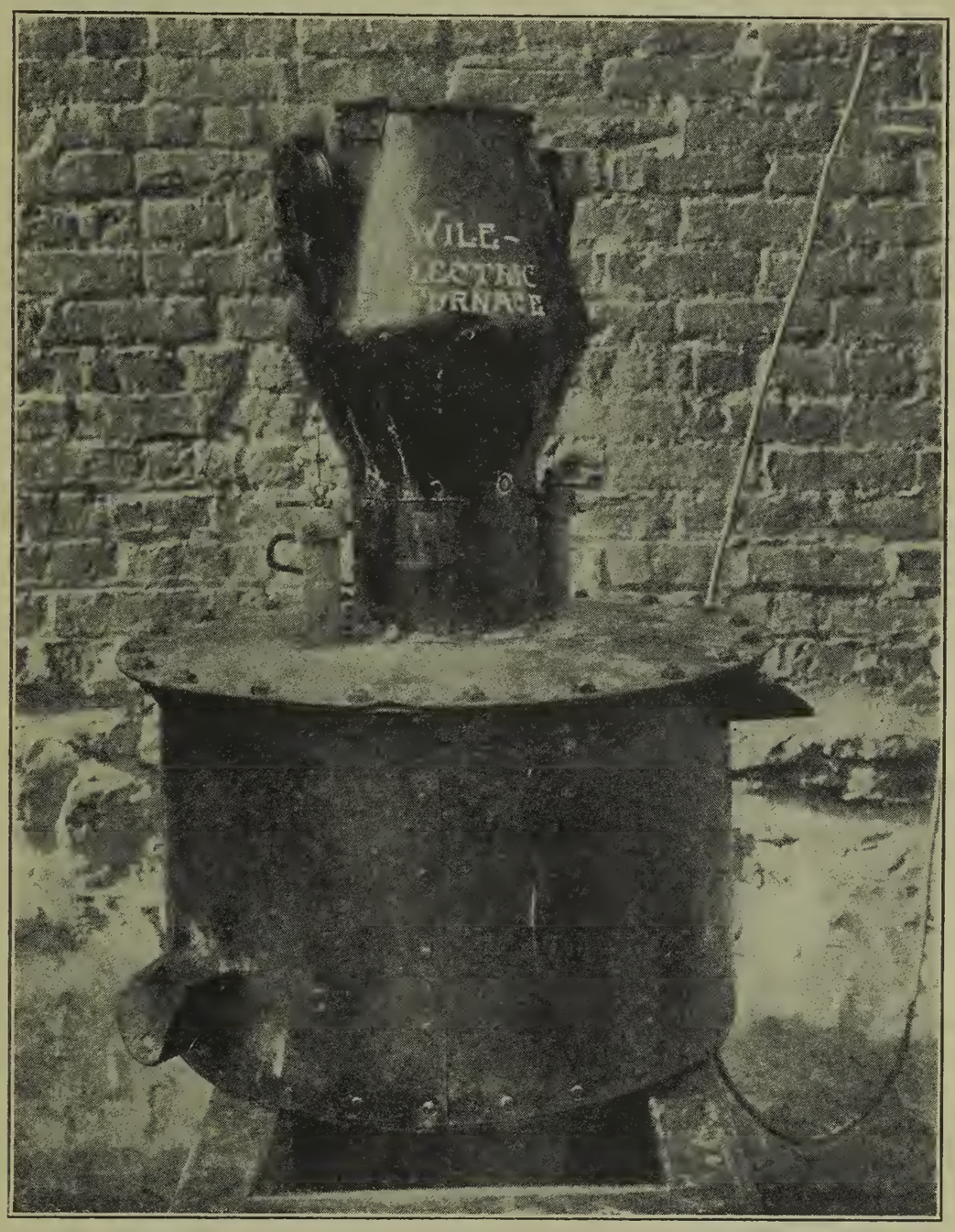

Fig. 7.

This furnace has a number of advantages over the commoner methods of smelting tin dross. In the older way soda ash is used for fluxing purposes, which being basic in character does not allow the slagging off of the iron or copper unless the silica is picked up from the side walls and bottom; the charge being in

a reverberatory furnace is subjected over a large area to the heat passing over it, causing a large flue loss. The resultant product must be refined to free it from iron, zinc or copper, and there is a large by-product known to the trade as hard sweats which must be put through a special treatment.

In this furnace the charge is above the slag; hence there will be no flue loss, as the cold charge will condense any volatile metals or oxides, and all gases from the furnace are run through water. The slag being composed at the start of 66.6 percent silica, 16.6 percent soda, and 16.6 percent lime, and the percentage of silica remaining the same, the oxides of iron, zinc and copper are slagged off as silicates, while at the temperature of the furnace, which is maintained at 1,650° C. or above, the tin will not combine with the slag, so that the ultimate tin loss in the slag is about 0.5 percent of the tin in the charge and the total loss is generally below 1 percent and never over 1.5 percent.

The power consumed varies, of course, with the amount of tin in the charge as well as the iron, zinc or copper to be slagged off, but for impure drosses varies between 600 and 900 K. W. hours per ton of tin produced. The cost of power is more than offset by the saving in tin, as the loss in the reverberatory is never under 7 percent and rarely that low.

It is possible to smelt tin drosses in the open hearth, thus permitting the use of the acid slag, but the loss in tin is very large because of the intense heat unless an expensive bag house is installed. In very few locations are there conditions warranting a smelter of this class of material to install a furnace of these proportions, as the cost of the gas, together with loss of values, and initial cost are far greater than electricity loss and initial cost in the electric furnace.

It has often been asked whether too much carbon will clog up the furnace, and while this is true in many furnaces, in this type the excess will flow out with the slag through the slag tap hole at the top of the bosh.

The life of the electrodes is about two months, as these do not come into contact with the air or charge, and merely dip into the slag, the rest of the electrode being protected.

It is possible to pick up the current in this type of furnace after

a shut-down of three hours, but the slag must be drawn for any longer delays than that.

The exhibition was followed by a long discussion which was concluded by Mr. Wile agreeing to a suggestion by Mr. G. A. Roush that he prepare a paper on his furnace for presentation at the next meeting of the Society.

COTTRELL PROCESS.

P. E. LANDOLT: I assume you are all more or less familiar with the fundamental principles of the process. Low tension alternating current is transformed to high tension of from 15,000 to 20,000 volts up to 100,000 volts, sometimes, and is then rectified by means of a synchronous switch. The resultant high tension direct current is transmitted from the rectifier to the high tension system which is usually a series of wires which are suspended in grounded pipes. The alternating current employed may be either 25 or 60 cycle. There seems to be no apparent difference in the efficiency of precipitation by means of the high tension produced from each source. A uniform gas flow is maintained and according to the voltage used we may use a corresponding size of pipe. We have in this experiment a 6-inch-diameter pipe and will probably develop about 35,000 volts. Usually in commercial practice we operate at 60,000 and 70,000 volts, and use a 14 or 18-inch pipe, depending on the character of the problem. Differences are found from the condition of the gas; namely, its temperature, moisture, acid content and the character of the dust content. In many cases the fumes are non-conducting; one case where it is particularly conductive is in sulphuric acid mist.

JOS. W. RICHARDS: Which do you operate with, the negative wire or the positive wire?

P. E. LANDOLT: Sometimes we operate with the wire negative and sometimes with the wire positive. We find the negative generally gives better results, that we can maintain a higher potential with the negative than with the positive, and get a better precipitation. The corona, which is produced on the negative, is not as powerful as on the positive.

G. A. ROUSH: What is the chemical nature of the material you expect to work today?

P. E. LANDOLT: We have taken some zinc dross from the resistance furnace. (See Fig. 8).

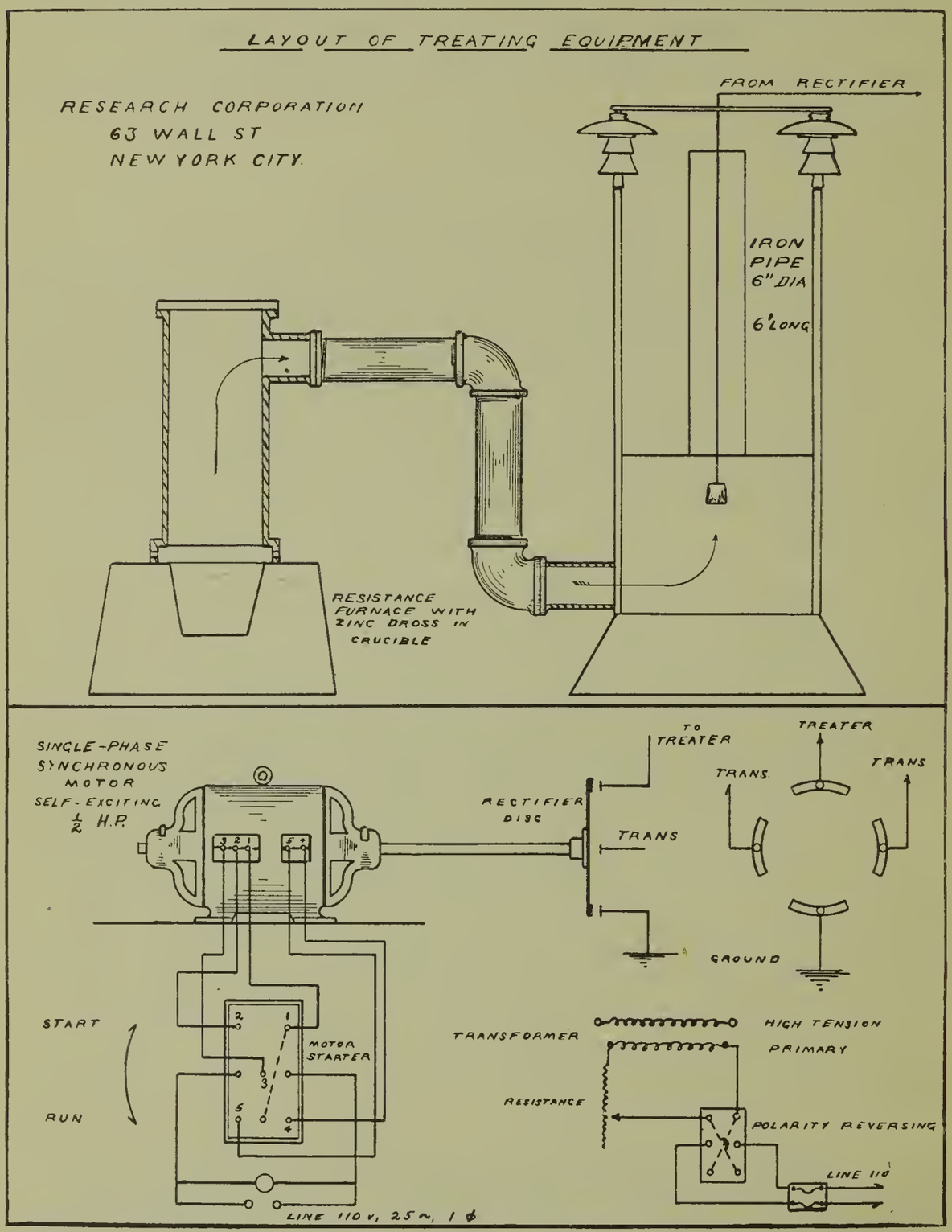

FIG. 8.

F. A. LIDBURY: I think the difficulties which Mr. Landolt temporarily experienced in his demonstration were too great a rate of flow of gas and too large a quantity of fumes for the

capacity and voltage at which he was running his apparatus. This demonstration is very instructive, because in talking to Mr. Bradley some days ago in regard to this method of fume precipitation he emphasized the extreme difficulties which have been met in several cases of the commercial application of this method of fume precipitation. These difficulties were largely in connection with the collection of proper data in regard to the amount of fumes per unit volume of gas and the rate of flow of the gas; and they have got to a point now, as I understand it, where they simply refuse to take any calculations on these data from possible clients, and insist upon making the measurements themselves, since the design of the apparatus depends to such a vital extent upon accuracy in these respects; so that the object lesson which was perhaps unavoidably given this afternoon was really a most instructive one, apart from the subsequent success of the precipitating operation.

Jos. W. Richards: Will Mr. Landolt kindly enumerate the different kinds of fumes to which this process is now commercially applied?

P. E. Landolt: Fumes delivered from slime furnaces in connection with the recovery of the gold and silver values from electrolytic copper refinery. The recovery of cement dust in connection with cement manufacture. The recovery of tin fumes in the form of stannic oxide; and zinc oxide from the brass industry. Also in lead fumes from the converting of lead copper matte as applied at the Garfield Smelter, at Garfield, Utah. The lead sulphides from the roasting processes, particularly from sintering machines, as applied at Tooele, Utah. Also in ventilation problems, such as the recovery of any kind of fine dust in the air of a building, for instance, bleaching dust, or carborundum dust, or alundum dust. Another case is borax dust. Also in the case of sulphuric acid. Sulphuric acid was the original application. We use it in the case of sulphuric acid mist. In that connection I will say that at present we are working on the removal of dust between the burners and the hot tower in connection with sulphuric acid work—that is still in the experimental stage.

JOS. W. RICHARDS: Has it been applied to the removal of dust from blast furnace gases?

P. E. LANDOLT: In the near future we expect to be working on that problem, we have not done anything as yet.

ALFRED H. COWLES: Does it work more favorably at high temperatures of the gases?

P. E. LANDOLT: In the cement works they work in the neighborhood of 1,000° F. At present we are working in connection with the dust removal from sulphuric gases in sulphuric acid manufacturing, working in the neighborhood of 1,000°.

M. G. LLOYD: Does the temperature affect the efficiency?

P. E. LANDOLT: Not so far as I have found out. Corona is formed at lower voltage. It seems that the increased temperature corresponds to decrease in pressure. The density of the gas is less and seems to affect the corona formation and spark-over voltage. There is some relation between these factors which has not been completely worked out to date.

ALFRED H. COWLES: Would any large increase of hydration affect it?

P. E. LANDOLT: Not from the standpoint of precipitation.

B. L. GLASCOCK: Has the process been used in the precipitation of carbon particles in smoke?

P. E. LANDOLT: Some experimental work has been done on that, and the bulk of this has been done at Pittsburgh. We have a man stationed at the Bureau of Mines at Pittsburgh carrying on some investigations in this direction. There has been a large amount of work done on this problem but it is still in the experimental stage. That is the understanding I have of it.

There is another application I forgot to mention among the commercial applications. That is the detarring of illuminating gas.

M. G. LLOYD: I understand that the voltage required depends on the diameter of the stack. I would like to hear how the amount of the discharge, that is the coulombs they pass over, is controlled. Is the quantity of the discharge noted proportional to the amount of fumes?

P. E. LANDOLT: So far as we have found out, the quantity of discharge does not seem to be proportional to the quantity of fumes. If we put together a certain sized wire and a certain sized pipe, there will be a definite voltage and a definite current; in fact, the current seems to be about the same, in different size pipes, for any potential. There seems to be no relation between the current consumption and the speed of the gases. We soon get into a complicated field if we undertake the discussion of the relation of current and voltage to the speed of gases and the number of particles in the gas. I hardly think we have time to go into that here.

JOS. W. RICHARDS: The larger part of the resistance is close around the wire, so that it does not make much difference how large the tube is?

P. E. LANDOLT: There is another thing that plays a part there—there is some condenser action involving the capacity of the discharge electrode as compared with the plate. There is one peculiar thing that happens—we find the material built up on the wire as well as the plate. Originally this was thought to be due to friction, but we have proved that it is not friction. You can pass the gases indefinitely through the pipe at the same speeds and the deposits will be nothing as compared with the deposit due to the current. The action of the current tends to vibrate the wire. It is necessary, in practice, to rap the pipes to remove the dust on them.

ALFRED H. COWLES: Does the dust deposit in hard masses, as a crust, or is it flocculent?

P. E. LANDOLT: It is flocculent. If a fume is finer than 200 mesh, there is a tendency to agglomeration after precipitation, but there is not quite that packing effect you asked about.

JOS. W. RICHARDS: Apropos of the remarks you made, I understand you had difficulty in determining the flow of gas.

F. A. LIDBURY: In getting accurate data at the establishments where they have been installed.

JOS. W. RICHARDS: It is an uncertain and difficult matter to determine the flow of gases. Mr. G. K. Herzog made a remark the other day which led me to make this suggestion.

He said that the flow of water in the hydraulic tubes was being determined by the use of common salt which was being distributed into the flow and then the amount of salt in the water, determined afterwards, showed by the dilutions you get, the amount of flow, and in that way the amount of water flowing had been determined to an accuracy of one-tenth of one percent. Maybe it would be possible to take a gas, which does not exist normally in the gases, and feed it continuously for a short time and carefully measure the amount of this gas which is put into the gases under treatment, and measure the output a sufficient distance from the input, and determine the proportion of gas from the dilution, and thereby determine the flow and the amount of gas which has flowed.

P. E. LANDOLT: We have applied that idea, but normally we have to deal with such large quantities of gases in smelters particularly, that it is impracticable to introduce sufficient gas to get a determination. A boiler house could have ammonia introduced and the ammonia in the gas could be determined after the point of admission to the system. If there is a certain percentage of CO_2 it may be increased by the introduction of CO_2. This determination can be made on a small scale, but with large quantities of gases it seems to be impracticable. We usually deal with gases in tens of thousands of cubic feet per minute, or hundreds of thousands of cubic feet per minute.

CARL HERING: The question of the agglomeration of particles was referred to just now. That recalls an experiment I made a good many years ago. I took a large bell-shaped jar filled with tobacco smoke, introduced two terminals of the ordinary induction machine, what is called the friction machine, giving high tension electricity, and turned on the machine, and in a second or two the air in the jar was clear. I noticed the smoke had agglomerated into streams, as if a spider had woven a web inside the jar. It seems from that experiment that at least tobacco smoke will agglomerate in that way.

JOS. W. RICHARDS: Has anyone thought of the idea of applying this to the condensation of metallic vapors, such as zinc vapor in condensers, so as to assist in the condensation?

P. E. LANDOLT: I do not know of any such method, although I see no reason why such an application could not be made practical.

THE OXIDATION RATE OF ELECTRODE MATERIALS.

A. T. HINCKLEY: This method of measuring the oxidation rate of electrode materials is essentially the same as described by Mr. C. A. Hansen in 1909 (Electrochemical and Metallurgical

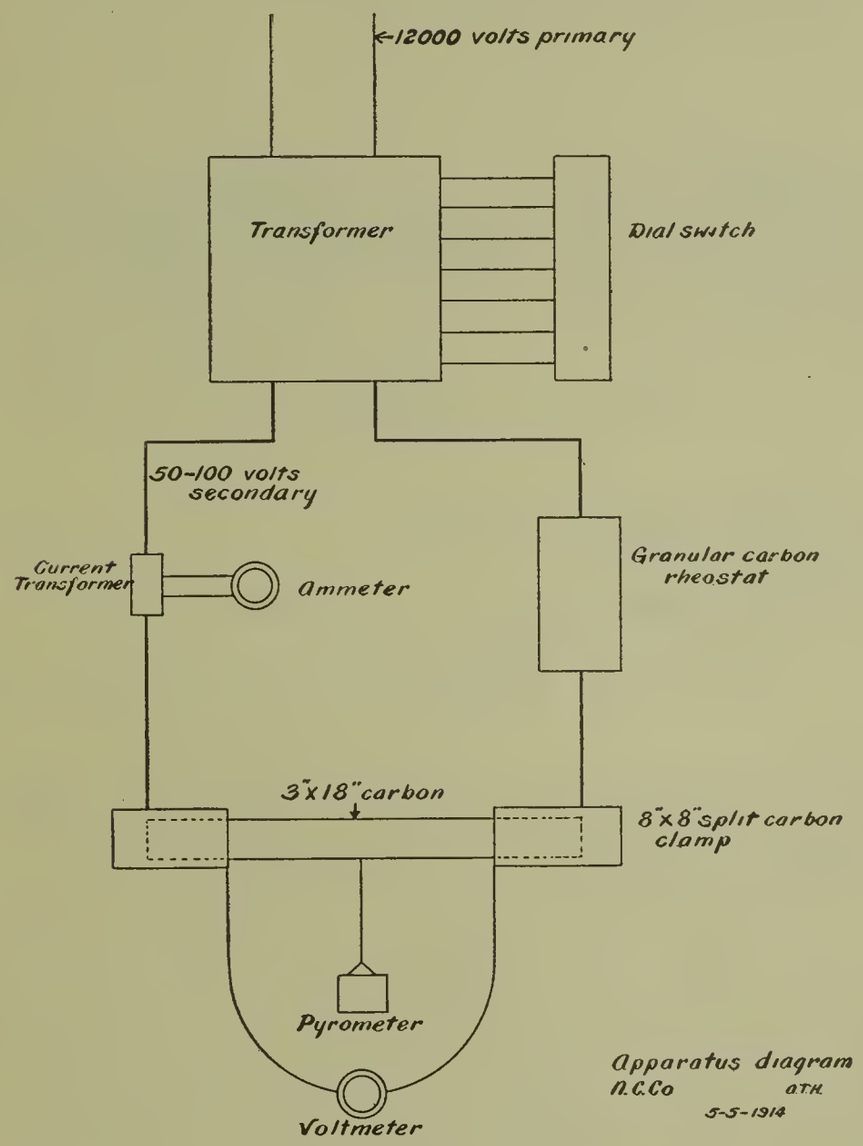

FIG. 9.

Industry, Vol. 7, page 389). We have found it necessary to use samples of larger diameter than one cm. when studying coarse grained carbons in order to secure uniform resistance. About 3" (7.6 cm.) proves to be a satisfactory size and the following diagram shows the electrical connections. On account

of the longer time taken to raise the large sample to, and cool it from, the desired temperature, it is necessary to determine the oxidation loss that occurs in each of these intervals and correct the total loss by that amount. (Fig. 9.)

This test may be carried out at constant temperature, constant current or constant potential. The constant temperature test has proven most useful in comparing different electrode materials, and electrodes manufactured by different methods. Owing to the different physical properties as real density, apparent density, etc., of electrodes made from different materials, it is difficult to determine which property to choose as the criterion, in selecting electrodes for comparison. The following carbons were selected on the criterion of *equal porosity,* and tested for rate of oxidation at 1,000° C.

Carbon	Rate of Oxidation
A	0.0050 g. per cm.^{2} per minute
B	0.0065 " " " " "
C	0.0075 " " " " "

Carbon *A* was regular graphitized stock of somewhat lower porosity than the average.

Carbon *B* was coarse grained amorphous carbon stock such as is used in large electrodes for steel furnace work.

Carbon *C* was average amorphous stock such as is used for graphitizing.

The rate of oxidation of electrodes varies very widely when other criteria are selected for choosing the carbons for comparison. Some electrodes of type *A* disintegrate rapidly with the "flaking" noted by Mr. Hansen, thus bringing their apparent oxidation rate above that of type *C*. This is important in determining the "life" of electrodes, but is not a true oxidation phenomenon. Slight variations in method of manufacture cause a variation in the oxidation rate of the resulting electrode, amounting to a change from 0.0065 to 0.0090 in the case of electrodes of type *C*.

The temperature at which oxidation starts may be very conveniently determined on samples set up in this way. This temperature varies considerably for different materials, but in general lies between 500 to 600° C. for materials of type *A* and between 400 and 500° C. for materials of type *C*.

TUBE FURNACE.

F. A. J. FITZGERALD: The tube furnace which is now running is worked on a different principle from that shown by Mr. Saunders, for the heat is not generated in the tube itself. The diagram shows the method of constructing the furnace which we find very useful for a great variety of experiments. Not only can this furnace be constructed cheaply, but it will last for a long time without repairs. It is obvious that the

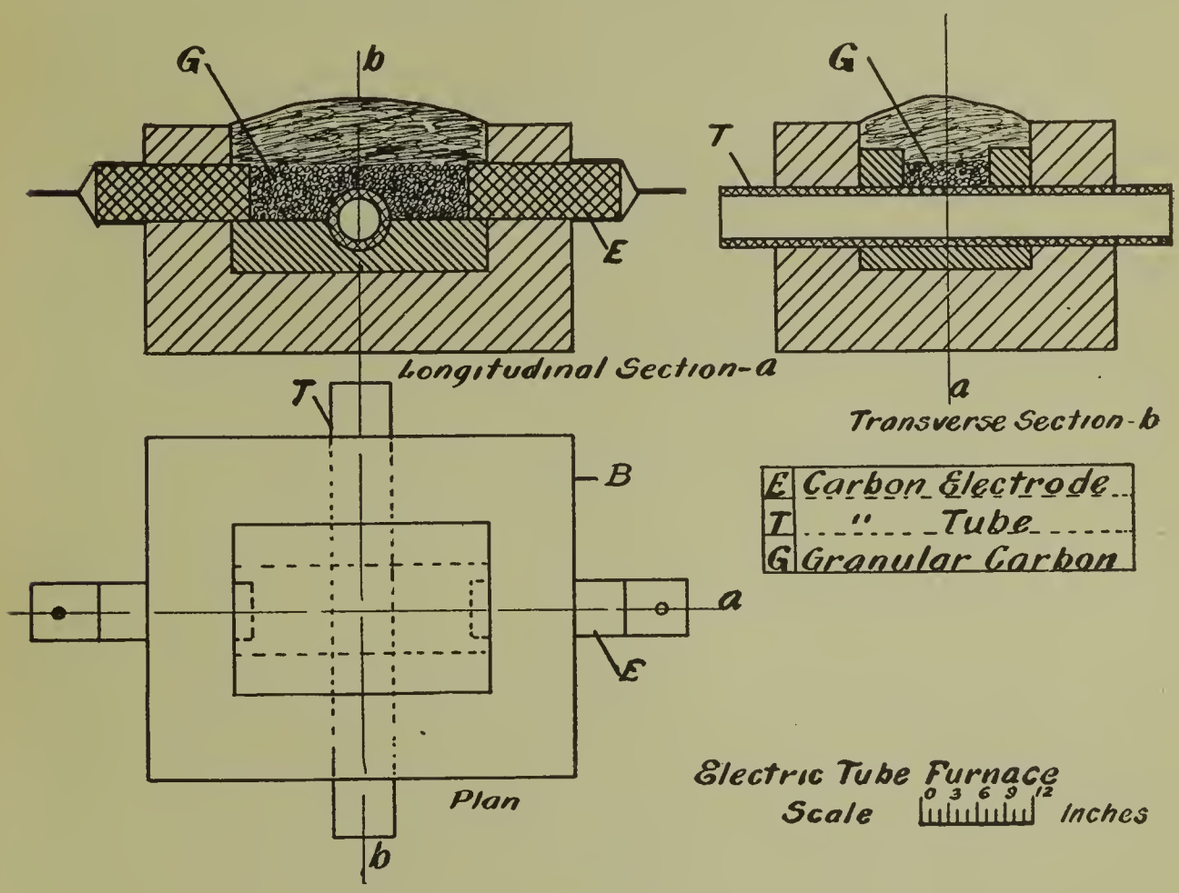

FIG. 10.

generation of heat occurs in the granular carbon which partially envelopes the tube.

The tube may be completely surrounded with granular carbon, but as shown in the sketch it only covers the upper part of the tube. Ours is constructed that way simply because we use it largely for melting point determinations with Seger cones, and in that case it is preferable to heat from above. (Fig. 10.)

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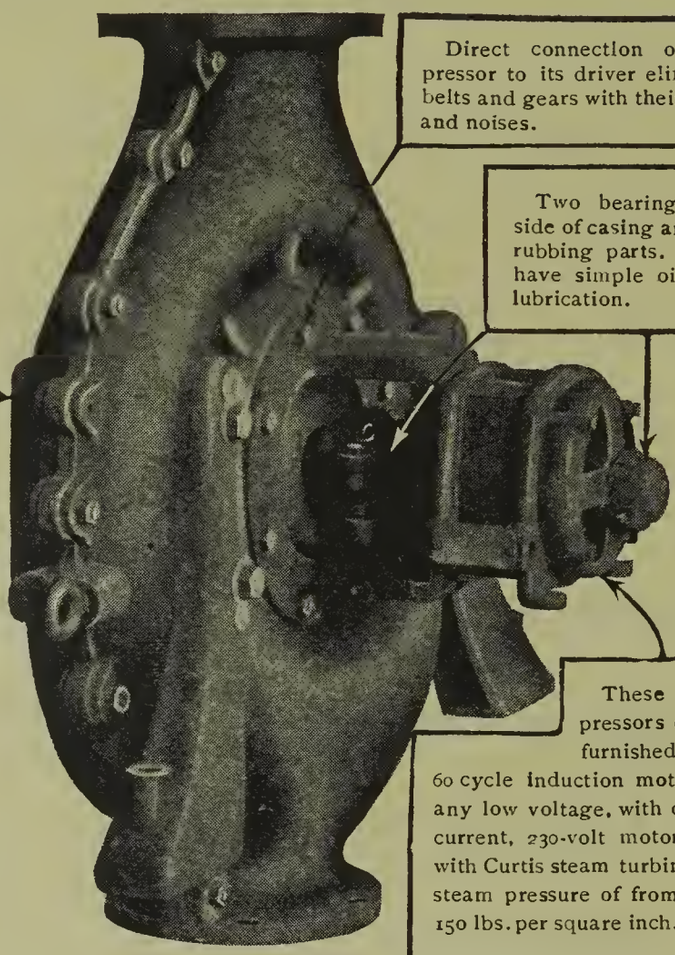

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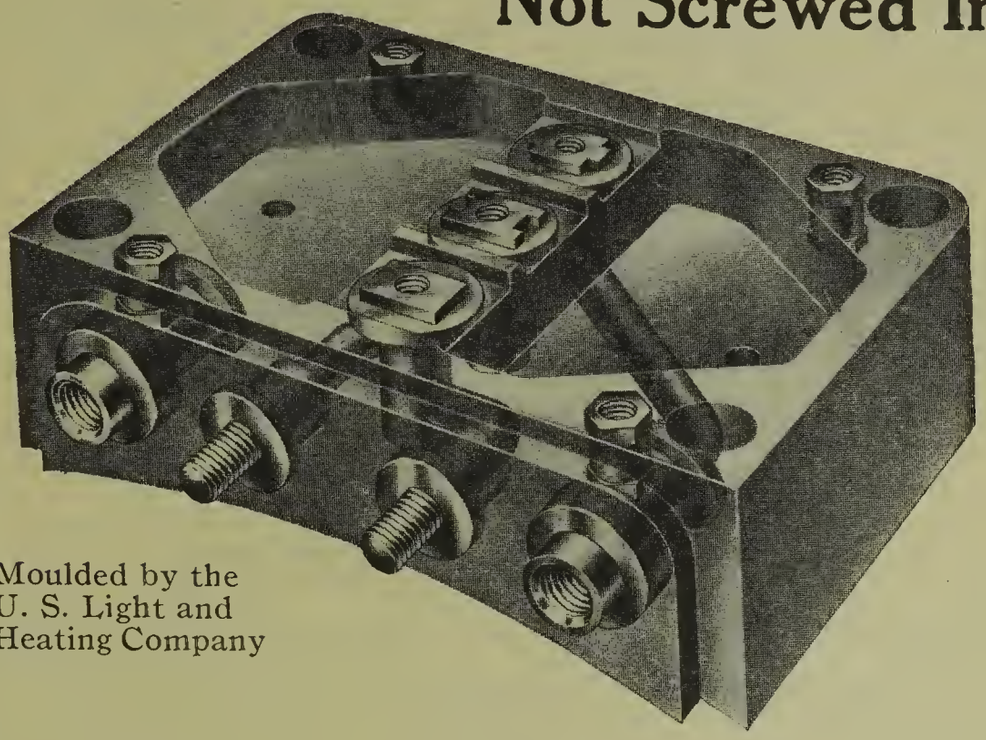

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